PRECIPITATE COARSENING DURING OVERAGING OF 2519 Al-Cu ALLOY: APPLICATION TO SUPERPLASTIC PROCESSING

by

WAYNE MATHÉ
Lieutenant, United States Navy
B.S., Mechanical Engineering, Tulane University, 1981

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ABSTRACT

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I. INTRODUCTION

Superplasticity is the ability of a material to exhibit very large neck-free tensile elongations prior to failure. Tensile elongations greater than 200% are generally considered indicative of superplastic behavior. Several materials have been processed to obtain extensions greater than 1000% [Ref. 1]. This high ductility in a superplastic material enables complex parts to be fabricated from a single piece of metal, thus eliminating the use of fasteners which would normally be required to join simpler shapes to make up the desired component. This reduction in the use of fasteners not only reduces weight, a factor vital in aircraft production, but also reduces susceptibility to fatigue and corrosion caused by the stress concentrations at fasteners. These advantages over standard engineering materials have generated much interest in both the commercial and military sectors.

Several different alloys are now being commercially produced using various processing routes. For aluminum alloys the two most widely used techniques are the Supral process and the Rockwell method. Both involve thermomechanical processing techniques designed to develop a fine grain structure and high-angle grain boundaries, necessary requirements for superplastic response. In the case of the Al-Cu-Zr Supral alloys, continuous

recrystallization is utilized to obtain a fine microstructure with high angle grain boundaries [Ref. 2]. Extensive warm working at 300°C followed by cold work is used in this process. Zirconium is added to the Al-Cu binary system to prevent recrystallization during the warm working phase. Dynamic recrystallization is achieved during the early stages of superplastic deformation (SPD). A temperature of 460°C is used for recrystallization and subsequent SPD [Ref. 1, 3, 4]. In the Rockwell process, the key to the thermomechanical processing sequence, developed for grain refinement of 7475 Al, is the use of particles to create nucleation sites for recrystallization [Ref. 5]. These particles are formed during an overaging step in the process and become nucleation sites as a result of warm rolling. Following a 482°C recrystallization heat treatment the particles redissolve. Both of the above processes will be discussed in greater detail in the background section of this report.

Due to the high recrystallization temperature for both processes, cavitation damage may develop during superplastic flow [Ref. 1]. The high Zr content of Supral increases cost of production and requires extra steps in the manufacturing process. In addition the extensive cold work in these processes may be undesirable or not possible in some alloys. The development of a simple process which would render a wider range of commercial Aluminum alloys superplastic without the aforementioned restrictions would be extremely valuable to industry.

Several years of research at the Naval Postgraduate School (NPS) have been devoted to the study of superplasticity in aluminum alloys and the development of a simple processing technique at a moderate temperature and without requiring any special alloy additions. Much success has been achieved with this process in the Al-Mg system and elongations exceeding 1000% have been obtained [Ref. 6].

The ultimate objective of this research is to utilize the background data compiled at NPS, on Al-Mg and Al-Mg-Li alloys, to obtain superplastic response in the commercial grade Al-Cu alloy 2519. Superplastic ductilities have been attributed to a combined precipitation and recrystallization mechanism in materials with large potential volume fractions of second phase. The grain refinement necessary for superplasticity has been reported to be a result of the combination of precipitation, recovery and PSN of recrystallization during TMP [Ref. 5]. It is hypothesized that a uniformly refined grain structure, suitable for superplastic response, is obtainable in the 2519 alloy by developing a uniform, high volume fraction of θ precipitates of a size large enough to act as nucleation sites for PSN during subsequent thermomechanical processing [Ref. 7].

In this study a commercial grade Al-Cu 2519 alloy was processed in an attempt to match the microstructure of the superplastic Al-Mg system using the techniques developed at NPS. In the first phase of this research a combination of cold rolling and overaging is used to produce uniformly

distributed θ phase particles of a size large enough for PSN. Previous research suggests a particle size of at least 1.5μm is necessary to act as future grain nucleation sites [Ref. 8]. In the second phase of this study the material in the overaged condition described above will be processed utilizing a TMP process similar to that developed at NPS for the Al-Mg systems. Metallography techniques will then be utilized to determine the overall effects on the microstructure and mechanical testing conducted to determine the extent of superplastic response.

II. BACKGROUND

A. SUPERPLASTICITY IN ALUMINUM ALLOYS

1. Grain Refinement

In order for a material to exhibit superplastic behavior, it must be capable of being processed into a fine equiaxed grain structure, normally of grain size in the range of 2 to 10μm and containing a large area fraction of high-angle grain boundaries [Refs. 1, 9]. There are several methods available for grain refinement in various alloys. Four general methods widely used are phase transformation, recrystallization, phase separation in duplex alloys, and inhomogeneous deformation of duplex alloys. Of these methods recrystallization is the most common technique for grain refinement and best suited for obtaining a fine grain structure in aluminum alloys [Ref. 5].

Two modes of recrystallization, continuous (CRX) and discontinuous (DRX), rely on particles to produce fine grain size. The effect of particles on recrystallizing grains depends on their size. Small particles, less than 1μm in diameter, suppress nucleation and retard recrystallization by pinning dislocations, subgrain boundaries and boundaries of recrystallizing grains. Large particles, greater than 1μm in diameter, may promote nucleation by creating nucleation sites for recrystallizing grains. Continuous recrystallization

has been used for grain refinement in superplastic Al-Cu-Zr alloys [Refs. 5, 10, 12].

When a fine-grain structure suitable for superplastic response has been produced, it must be maintained during the superplastic forming process at elevated temperatures. One way to keep grains from coarsening during the forming process is to use solute atoms and particles to exert a drag force on the grain boundaries, thus restricting grain growth [Ref. 5].

2. Processing for Superplasticity

In order to obtain a microstructure suitable for superplastic response thermomechanical processing techniques have been developed and utilized. The two most common processes used in industry to obtain superplastic deformation in high strength aluminum alloys are the "Rockwell" method and the "Supral" process.

The thermomec[illegible] grain refinement in 7475 aluminum, developed at the Rockwell International Science Center, will be discussed first. The key to this processing technique is to use a high density of micrometer-size particles to act a nucleation sites for recrystallizing grains [Refs. 5, 11]. The process involves four primary steps and is illustrated in Figure 2.1. Parameters for the process are presented in Table 2.1.

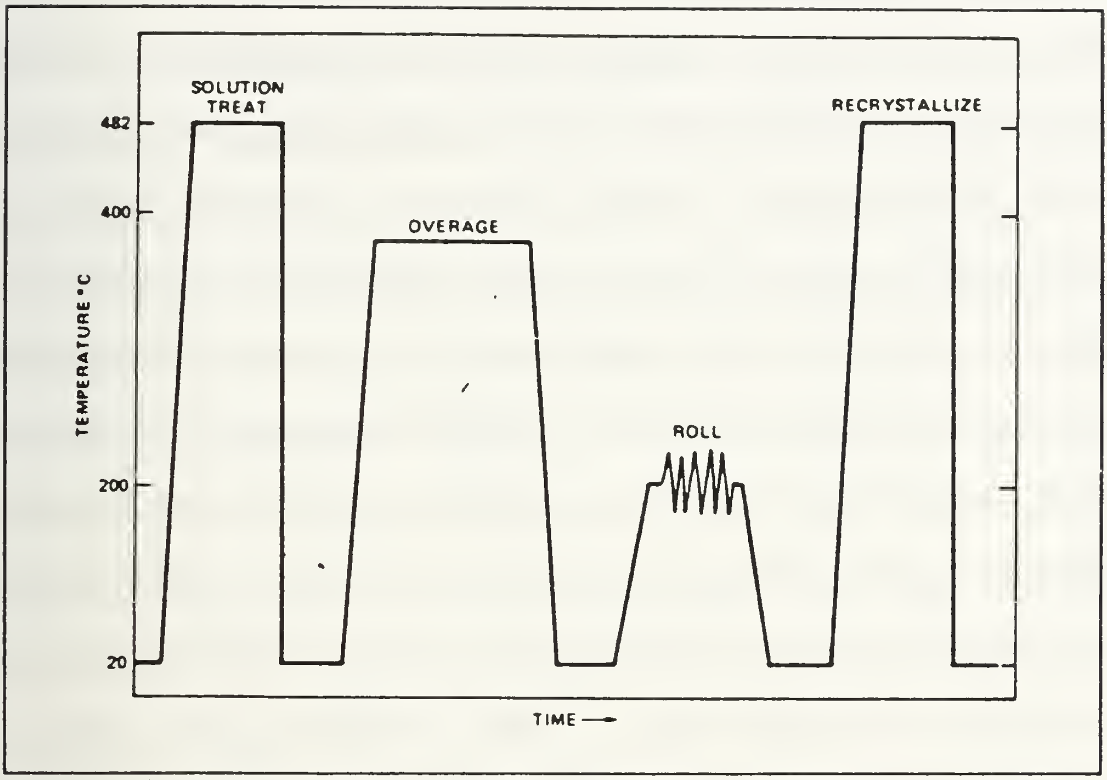


Figure 2.1. Schematic of the thermomechanical processing sequence used for grain refinement of 7475 Al [Ref. 5].

TABLE 2.1 THERMOMECHANICAL PROCESS PARAMETERS FOR GRAIN REFINEMENT OF 7475 Al [Ref. 5].

Step	Parameters
Solution Treatment	482°C, 3 hours, water quenched
Overaging	400°C, 8 hours, water quenched
Rolling	200°C, 90% reduction
Recrystallization	482°C, 0.5 hours, water quenched

In step 1 a solution treatment is utilized as a starting condition, dissolving precipitates and placing the Zn and Mg solutes into solution. Step

2 of the process is an overage at 400°C for 8 hours followed by a water quench. This produces equilibrium M-phase particles larger than 0.75μm which affect nucleation of recrystallizing grains. Next the 7475 is warm rolled at 200°C to a 90% reduction which introduces defects into the alloy essential for recrystallization. The final step is a high temperature recrystallization at 482°C for 30 minutes which allows time for the recrystallizing grains, approximately 10μm in diameter, to consume the entire matrix and the precipitate particles to redissolve. It should be pointed out that the rate of heating to the recrystallization temperature in this step is a critical aspect of the process. There is a remarkable increase in the recrystallized grain size if the heating rate in this step is slowed, therefore a rapid heating to recrystallization temperature is required for a finer recrystallized grain structure [Ref. 5].

A second thermomechanical processing technique was developed by British Aluminum [Ref. 5, 12, 10] (now British Alcan Aluminum plc) to produce a medium-strength superplastic Al-6%Cu-0.4%Zr (Supral) alloy. The underlying principal employed in this process is to add a third alloying component, in this case Zirconium, to an Al-Cu system in a way which enables uniform, fine-scale $ZrAl_3$ particles to be precipitated from solid solution. These precipitates which remain in the form of second-phase particles at high temperatures, i.e. temperatures where superplastic forming is possible, play a vital role in restricting recrystallization and grain growth. They have been

determined experimentally to hinder both recovery and recrystallization and increase the temperature for the transition between continuous and discontinuous recrystallization [Ref. 12]. A high Zirconium content (approximately 0.4%), which by far exceeds its solubility in aluminum, is essential to this processes ability to obtain superplastic response. It also causes the greatest problem to overcome in making alloys of this type by creating a requirement to suppress the separation of coarse particles of Al_3Zr during casting. To achieve the degree of supersaturation with zirconium, solidification must take place under non-equilibrium conditions and rapidly enough to prevent or at least minimize separation during solidification. Therefore, to manufacture Supral alloys a specially designed casting system must be employed which minimizes the residence time of molten metal in the sump, achieves solidification as rapidly as possible but does not result in high residual stresses in the casting [Ref. 13]. The four-step process for Supral 220 is illustrated in Figure 2.2.

In step 1 of the Supral process, a homogenization is conducted. The ingot is held at a sufficiently high temperature to allow diffusion to take place thus removing compositional variations and dissolving coarse intermetallic compounds that separate during solidification. The high level of zirconium supersaturated in the casting, together with its low solid solubility in aluminum, means that any elevated temperature treatment would result in precipitation of zirconium from the supersaturated solid solution. Therefore,

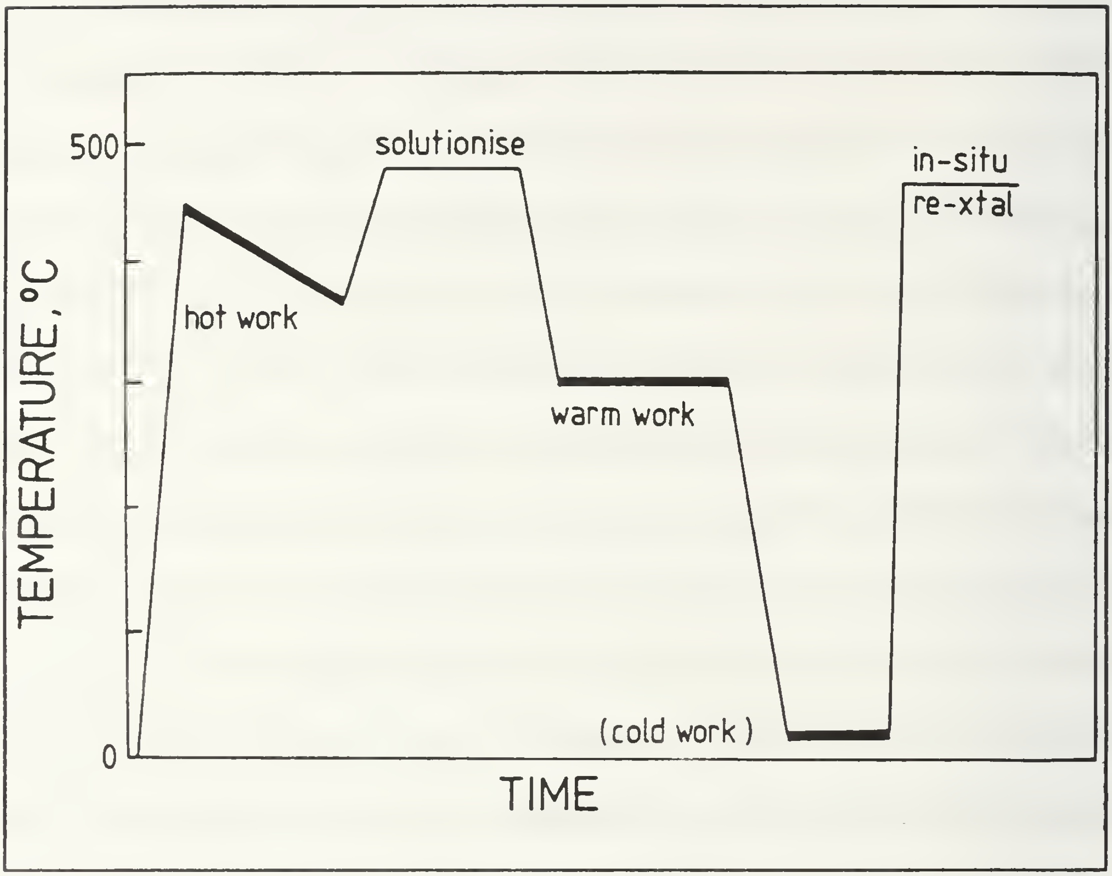


Figure 2.2 Schematic of the processing route for Supral 220 used to generate a fine grain microstructure by "dynamic recrystallization [Ref. 1].

a conventional, high temperature solution treatment would result in the formation of a relatively coarse dispersion of Al_3Zr particles that would not be very effective in controlling grain growth during superplastic deformation. The control of the dispersion and size of these grain-pinning particles is very sensitive to temperature and time during this heat treatment portion of the process. The hot rolling procedure used during this phase of the process is no different from that employed for conventional strong aluminum alloys although precise control of temperatures and times is required to ensure no excessive

growth of the Al_3Zr particles [Ref. 13]. The next step of the process is to warm work the material. This is accomplished by heating in an air furnace at 300°C and rolling to a 90% total reduction at 20% per pass [Ref. 12]. During this warm working, recovery and recrystallization is prevented by the $ZrAl_3$ particles and to a lesser extent by the copper and magnesium in solid solution [Ref. 1]. After a quench to room temperature the material is heavily cold worked to introduce a high strain energy prior to subsequent recrystallization. This step is then followed by reheating to the solutioning temperature where dynamic recrystallization occurs during the superplastic forming operation [Ref. 1].

B. PREVIOUS RESEARCH AT NPS

1. Al-Mg Studies

Initial studies at the Naval Postgraduate School on the Al-Mg system were concerned with developing a uniform distribution of the β phase precipitate. Work was concentrated in developing a thermomechanical process which would eliminate β phase precipitation at the grain boundaries thus decreasing susceptibility to intergranular attack [Ref. 14]. During this process development it was discovered that warm rolling of an Al-10%Mg-0.5%Mn alloy at 300°C resulted in a refined structure stabilized by a dispersion of β (Al_8Mg_5) and $MnAl_6$ particles. This structure produced by warm rolling was superplastic at a relatively low temperature, 300°C, and high

strain rate, $\dot{\varepsilon}$~$2x10^{-3}s^{-1}$ [Ref. 15]. Additional testing of the TMP in an Al-10%Mg-0.1%Zr material showed similar success. The same TMP produced an even more superplastic response in an Al-8%Mg-1%Li-0.15%Zr alloy [Ref. 16]. The application of this TMP on 7475 Aluminum, which is presently made superplastic by the Rockwell process, to assess the degree of grain refinement, was conducted by Lee and McNelley. Elongations up to 280% at 300°C without cavitation, were achieved [Ref. 17].

2. NPS TMP for Superplasticity in Aluminum Alloys

a. Process Description

After several modifications, as determined necessary by additional studies, the most recent NPS TMP for Al-Mg alloys, consists of an initial solution treatment with subsequent hot working (upset forging) followed by an oil quench to room temperature. The material is then warm rolled at 300°C, a temperature below the β phase solvus temperature, with a 30 min anneal between each rolling pass (Figure 2.3). The most recent rolling schedule developed insures both strain and strain rate increase on each successive pass (Table 2.2) [Ref. 18].

TABLE 2.2 Al-Mg ROLLING SCHEDULE [Ref. 18]

PASS #	h_i (in)	Δh (in)	ε_p (%)	$\dot{\varepsilon}_p$ (s^{-1})
1	1.00	0.10	16.2	0.893
2	0.90	0.11	12.2	1.045
3	0.79	0.1[illegible]	13.9	1.196
4	0.68	0.11	16.2	1.399
5	0.570	0.11	19.3	1.680
6	0.460	0.11	23.9	2.104
7	0.350	0.085	24.3	2.435
8	0.265	0.07	26.4	2.931
9	0.195	0.055	28.2	3.547
10	0.140	0.040	28.6	4.219
11	0.100	0.030	30.0	5.130
12	0.070	0.023	32.9	6.464

The initial solution treatment ensures full homogenization of the material before the start of the processing. The warm working sequence, below the β phase solvus temperature, results in concurrent precipitation of intermatallic β and formation of a subgrain structure in a matrix of elongated grains [Ref. 19]. The 30 min anneal between each rolling pass is required to allow for the appropriate precipitation and to facilitate the static recovery processes [Ref. 20]. The full details of microstructural transformation during the sequence of warm rolling and annealing cycles are not fully understood, but the size of the fine grain structure achieved

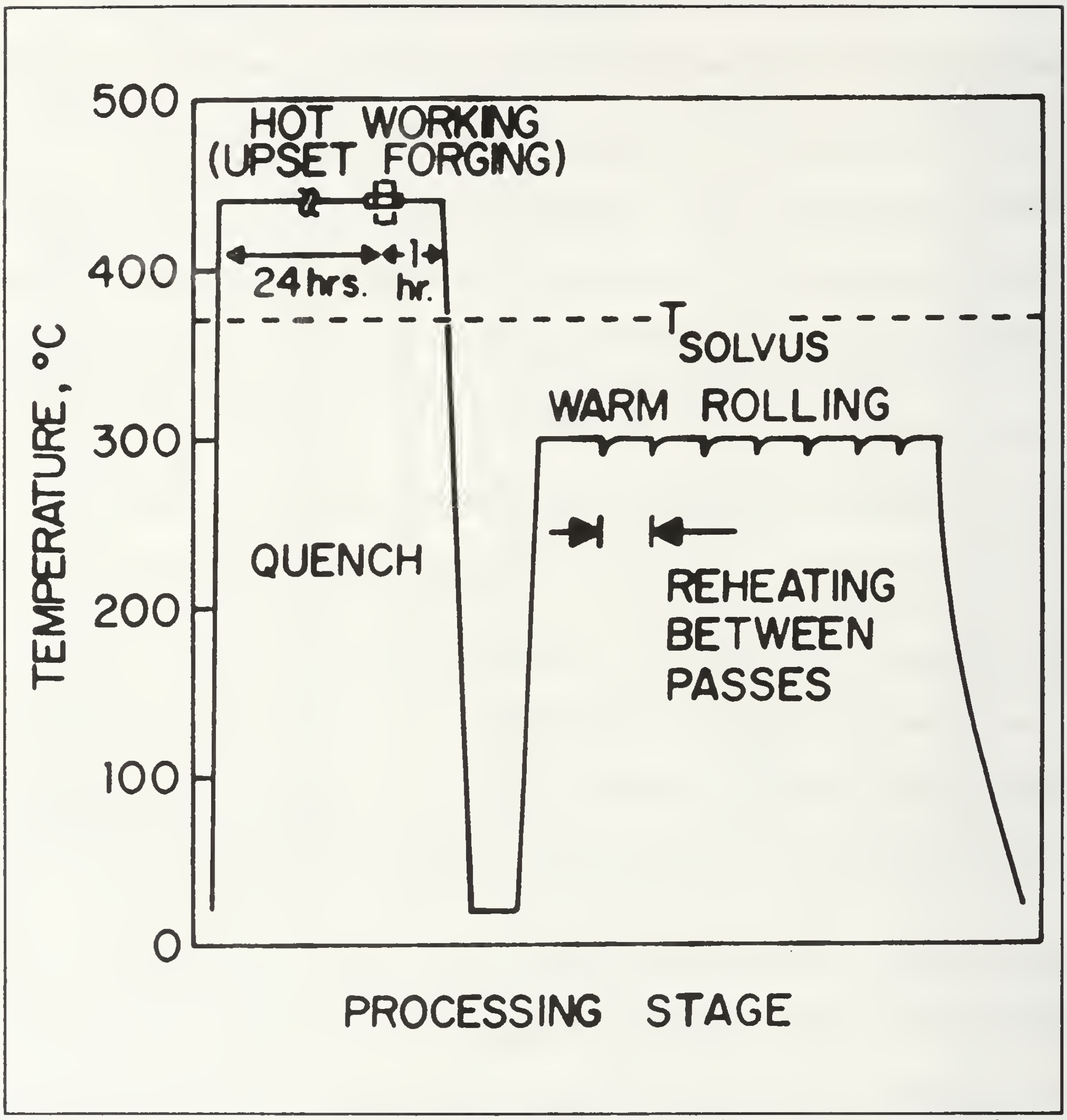


Figure 2.3 TMP Schedule. Homogenization and quenching, followed by 12 rolling passes with a 30 minute anneal at 300°C between each pass. [Ref. 18]

corresponds approximately to the interparticle spacing which suggests the occurrence of particle-stimulated nucleation (PSN) during annealing [Ref. 7]. Research in progress has shown grain nuclei in association with particles during the reheating anneals. The increase in rolling strain enhances the

superplastic ductility of the alloy by creating a more uniformly refined grain structure (3-5 m) during the recrystallization process [Ref. 21]. The latest experimental testing shows microstructural evidence that in the early stages of the TMP, precipitation reactions dominate. In the later stages PSN of recrystallization competes with recovery as the predominate factor governing microstructural evolution [Ref. 18].

In some respects the NPS process represents a combination of the Supral and the Rockwell process. The primary difference however, is the second phase particle size and how it is exploited in the process. The β-phase particle is on the average twice the size of the M-phase particle in 7475. This larger size may produce greater localized strain and therefore the extensive cold working of the Rockwell process is not required. In addition, unlike the Rockwell process where the second phase is redissolved at the high forming temperature the Al-Mg β particle remains a part of the structure throughout the superplastic forming process.

b. Advantages Over Current Industry Processes

The industrial superplastic processes addressed previously require high temperature, approximately 90% of aluminum's melting temperature, to obtain superplastic flow. Cavitation, which is the formation of internal porosity during plastic deformation at these high temperatures, can be a serious problem. In addition the low strain rates required present the problem of lowering production rates [Ref. 6]. The addition of special alloying

elements to the material to promote superplastic microstructure formation increases the cost of production and complicates the process. Finally, extensive cold working may not be practical for some materials.

Therefore, the advantages of the NPS TMP over the processes already addressed is its ability to obtain superplastic response by a relatively simple process, which does not require a high temperature recrystallization treatment.

This TMP method which incorporates PSN for recrystallization may facilitate attainment of grain sizes in the range of 3-5μm in high strength aluminum alloys [Ref. 17].

C. WROUGHT ALUMINUM ALLOYS

Aluminum alloys are attractive materials for commercial use for a wide variety of reasons. They have excellent strength-to-weight ratios, are corrosion resistant, can be fabricated easily and have excellent physical and mechanical properties. Aluminum alloys are divided into two major categories. Casting compositions which are designated by a 3 digit system followed by a decimal (e.g., 2xx.x), and wrought compositions which are shaped by plastic deformation and designated by a four-digit system (e.g., 2xxx). These two main groupings are further divided into two subgroups, heat treatable and non-heat treatable [Ref. 22].

For wrought aluminum alloys, the type used in this research, the first number indicates the principle alloying element. The 2 in the 2519 system indicates copper is the principle alloying element. The second digit indicates the alloy modification, i.e. the fifth modification to the original alloy. The last two digits serve to identify the different aluminum alloys in the group. Temper designations generally follow the alloy designation number and are used to identify how the alloy has been heat treated, strain hardened, annealed, etc. [Ref. 22].

1. Aluminum-Copper System

The 2xxx series alloys require a solution heat treatment to obtain optimum properties. In this condition mechanical properties are similar to those of low carbon steel. Precipitation heat treatment (aging) is often employed to further increase mechanical properties. Alloys in this series are well suited for structures requiring high strength-to-weight ratios and are commonly used to make aircraft fuselage, wing skins and structural parts [Ref. 22]. The binary Aluminum-Copper Phase Diagram is shown in Figure 2.4 [Ref. 23].

2. Strengthening Mechanisms

The objective of strengthening mechanisms in the design of aluminum alloys is to increases strength, hardness and resistance to wear, creep, stress relaxation, or fatigue [Ref. 22]. There are several strengthening

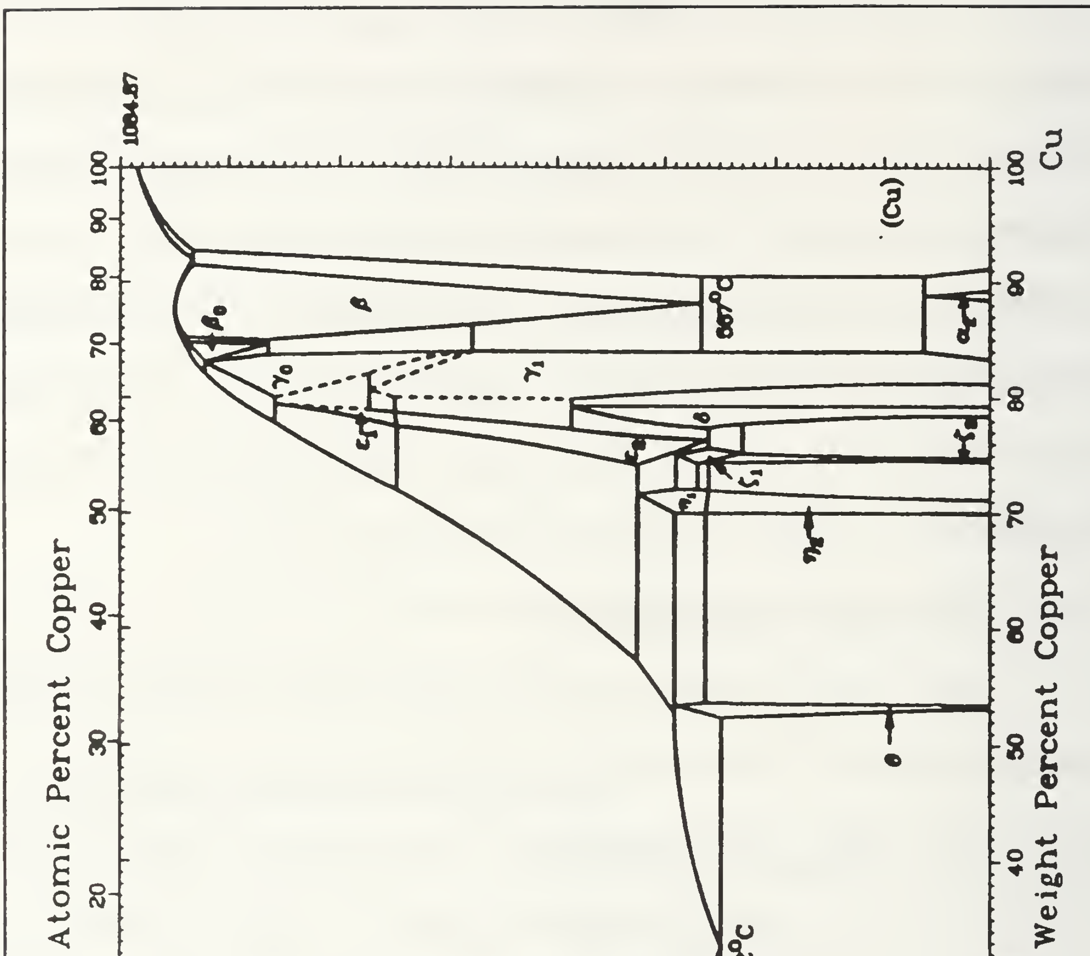


Figure 2.4 Al-Cu Phase Diagram [Ref. 23].

mechanisms available to enhance these mechanical properties of pure aluminum. It is necessary to understand how they contribute to the system to achieve these optimum properties and thus how the alloy used in research will be affected by these mechanisms. Slip is due to the motion of dislocations through the crystal lattice. This process provides ductility in metals, which is desirable, but easy dislocation movement will cause the material to be to weak and not suitable for engineering purposes. The mechanical properties of a metal or alloy can be controlled by interfering with this movement. The essential goal of strengthening therefore, is to impede the motion of these dislocations. Various mechanisms for achieving this in the Al-Cu 2519 system are addressed below.

a. Solid Solution Strengthening

The introduction of point defects, in particular substitutional and interstitial atoms by addition of an alloy to a pure material, disturb the atomic arrangement in the lattice and interfere with the movement of dislocations, or slip. The degree to which strengthening occurs depends on two factors. First, the larger the size difference between the original atom and the added atom, the greater the increase in the strengthening effect. Second, the larger the addition of the alloying element the greater the strengthening effect. That is up until the point where the solubility limit is exceeded, at which point a different strengthening mechanism, dispersion strengthening is produced [Ref. 24]. Solid solution strengthening is an important strengthening

mechanism for almost all aluminum alloys. In the Al-Cu 2519 system studied in this research the addition of 6% Copper, with an atomic radius 12% smaller than that of Aluminum [Ref. 24], along with Magnesium and Manganese, is used to take advantage of this mechanism.

b. Precipitation Hardening (Age Hardening)

Age hardening provides dispersion strengthening through heat treatment. This type of strengthening arises when the solubility limit is exceeded in an alloy, thus resulting in formation of a second phase. The atomic arrangement at the boundary between the two phases is not perfect and thus interferes with the slip of dislocations which acts to strengthen the material. The larger the amount of precipitate the greater the increase in strength of the alloy [Ref. 24]. A three-step process is utilized to produce a uniform dispersion of a fine, hard coherent precipitate in a softer, more ductile matrix. In the first step, a solution treatment, the alloy is heated above the solvus temperature to dissolve any second phase and produce a homogeneous single-phase solid solution. Immediately following solution treatment the alloy is quenched. This rapid cooling does not allow time for the atoms to diffuse to potential nucleation sites and the structure still contains only one phase, which is now a supersaturated solid solution (SSS) containing excess alloy atoms. This structure is not in equilibrium. In the third step the SSS is heated to a temperature below the solvus where the atoms are able to diffuse to form the equilibrium $\alpha+\theta$ structure. This part of the process is temperature-time-

dependant and different alloys have very different requirements to reach peak strength [Ref. 24].

In the specific alloy used in this research Al-Cu 2519, which has a copper content greater than 5.65%, complete solution can never occur since solution treatment above the solvus line is not possible or melting will occur (Figure 2.4). Therefore, the temperature used for solution treatment is as close to the eutectic temperature of 548°C as practical, while providing a margin of safety [Ref. 25].

In the aging of Al-Cu alloys, a continuous series of precipitates forms before the equilibrium θ phase is produced. In the early stages of the aging process localized concentrations of copper atoms form on {100} planes in the α matrix and produce very thin precipitates called Guinier-Preston, or GP-I, zones. As the aging process continues more copper atoms diffuse to these sites creating thin discs, or GP-II zones [Ref. 24]. These zones create strain fields that increase resistance to deformation, which is why in some alloys, specifically the 2xxx series, mechanical properties improve during natural aging and useful engineering materials can be obtained through natural aging alone. At higher temperatures the GP zones transition to θ" and θ' nonequilibrium, coherent precipitates (Al_2Cu), which produces the highest strength condition. For engineering applications, the useful aging temperature range for Al-Cu alloys is from 120 to 230°C [Ref. 22]. As time and temperature continue to increase the stable, noncoherent θ phase is formed, at which point

the alloy is said to be softened or "overaged" [Ref. 25] and is considered unusable in standard engineering applications. The stages of aging for the Al-Cu system are diagramed below:

$$SSS \rightarrow \alpha + GP\text{-}I \rightarrow \alpha + GP\text{-}II \rightarrow \alpha + \theta'' \rightarrow \alpha + \theta' \rightarrow \alpha + \theta\,(Al_2Cu)$$

c. *Strain Hardening (Work Hardening)*

Strain hardening is the strengthening of a material by increasing the number of dislocations by deformation or cold working [Ref. 24]. Strengthening occurs due to dislocation interaction directly among themselves or indirectly with stress fields caused by lattice defects. These interactions produce a reduction in the mobility of dislocations thus requiring an increase in the stress needed to move them [Ref. 26].

D. DIRECTION OF CURRENT RESEARCH

From the information obtained by previous research at NPS on the Al-Mg system, in particular the particle stimulated nucleation observed at the β particles [Ref. 7], an attempt is being made to match the superplastic Al-Mg microstructure in an Al-Cu system. It is hoped that by matching this microstructure superplastic response similar to that of the Al-Mg system will be obtained. It is the intent of this research then, to produce θ particles in the Al-Cu system to act as sites for particle-stimulated nucleation during the warm rolling process.

For this research a commercial grade 2519 Al-Cu alloy was chosen. In the initial phases, the kinetics for overaging of the alloy were studied to determine appropriate temperature time parameters to produce a θ phase of size and distribution similar to that of the β particle in the superplastic Al-Mg system. The scanning electron microscope (SEM) in the backscatter mode was utilized to determine particle size and distribution. In the next phase of this research, the thermomechanical process utilized to achieve superplastic response in the Al-Mg system was attempted on an overaged sample of the 2519 alloy. It should be noted that the TMP process was modified, from that used in the Al-Mg system, with a pre-warm roll overage. This was done due to determination during the overaging kinetics experiments that the θ phase particles would not have sufficient time to reach the size deemed necessary for PSN during reasonable warm rolling schedules alone. SEM and optical microscopy were utilized in an attempt to determine how successful the process had been in matching the microstructure of the superplastic Al-Mg system. In the follow on phases of this research tensile tests were conducted to assess the extent of subsequent superplastic response.

III. EXPERIMENTAL PROCEDURE

A. MATERIAL

The alloy used in this research Al-Cu 2519, was supplied by the ALCOA Technical Center. The as received material was a plate (Lot No. - 589681) 12 x 24 x 0.875 in. (304.8 x 609.6 x 22.2 mm), in a T87 temper (solution heat treated at 535°C, cold rolled (7%) and artificially aged 24 hrs at 165°C) [Ref. 27].

The material composition, listed in Table 3.1, is within the required specification limits for a 2519 alloy as specified in the Metals Handbook [Ref. 22].

TABLE 3.1 ALLOY COMPOSITION (WEIGHT PERCENT)

Cu	Mn	Mg	Fe	Zr	V	Si	Ti	Zn	Ni	Be	B	Al
6.06	0.30	0.21	0.16	0.13	0.09	0.07	0.06	0.03	0.01	0.002	0.001	bal.

B. OVERAGING PROCESS

Initially a 1.5 in strip was cut from one side of the plate. This strip was then divided into ten 1.5 x 0.875 x 2.4 in. (38.1 x 22.2 x 61.0 mm) billets. Five of these billets were then sectioned into 24 rectangular coupons each of average size 0.56 x 0.48 x 0.44 in. (14.2 x 12.2 x 11.2 mm), see Figure 3.1. This

size was used to minimize the amount of material consumed in testing while providing a sample large enough to work with. In addition this size allowed a minimum of three indenter diameters between hardness test indentations and between indentations and the edge of the coupon, as required by ASTM standard [Ref. 28]. These coupons were then solution treated and aged, with and without prior cold rolling, at various temperatures and times.

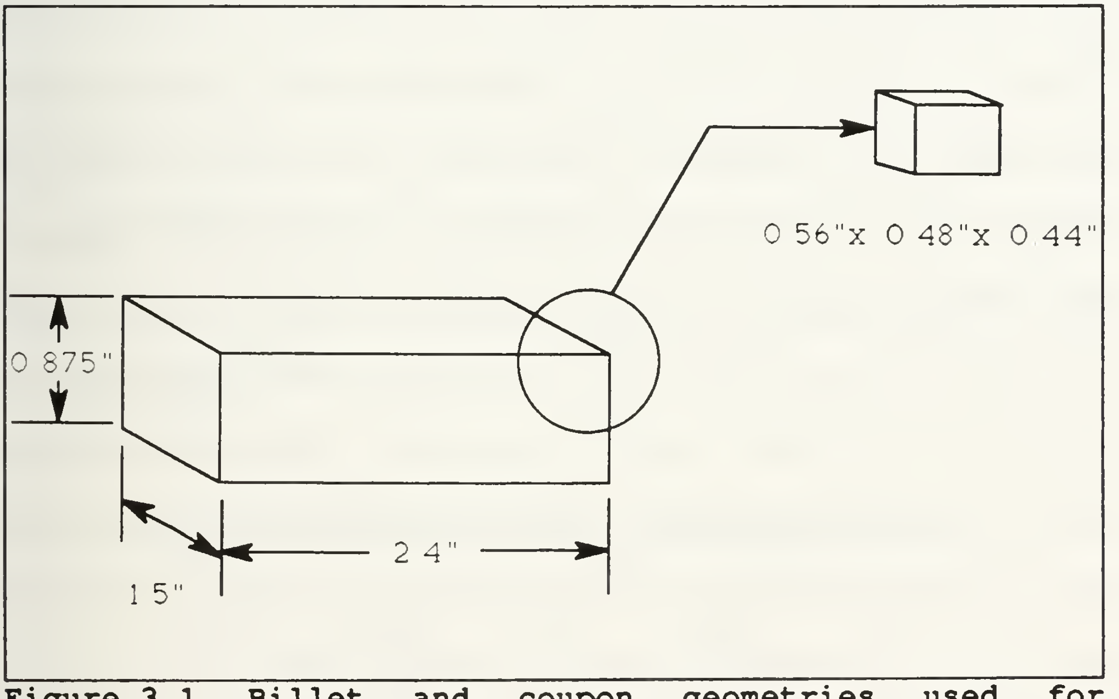


Figure 3.1 Billet and coupon geometries used for testing.

1. Solution Treatment

A uniform starting condition was obtained by solution treatment prior to any additional heat treatment or cold work. The solution treatment dissolves the soluble precipitates, i.e. places in solution the Cu and other alloying elements. The solution treatment was performed in an air furnace at 535°C for 100 minutes (soak time began when thermocouples recovered to original operating temperature) as recommended for the billet size used by the ASM Handbook Volume 4 [Ref. 25]. Due to the high Cu content of this alloy, 6.06%, it is solution treated at a temperature just below the eutectic temperature (Figure 2.4), therefore complete solution can never occur [Ref. 25]. To ensure the quench rate would be similar to that for the billet size used in later testing the coupons were put back together to form blocks then wrapped in aluminum foil and secured with nichrome wire before solutioning. Upon completion of the solution treatment the samples were quenched in room temperature water at about 22°C. This rapid cooling does not permit time for atoms to diffuse to potential nucleation sites creating a supersaturated solid solution alpha phase containing excess copper [Ref. 24].

2. Cold Working

In the 2xxx series, cold working of freshly quenched material greatly increases its response to later precipitation heat treatment [Ref. 25]. The introduction of dislocations during rolling at room temperature was designed

to provide nucleation sites for precipitating phases, increasing the number and homogeneity of their distribution.

3. Aging

Artificial aging is designed to produce a uniform dispersion of a fine, hard coherent precipitate in a softer, more ductile matrix [Ref. 24]. Artificial aging, in an air furnace, at various temperatures and times (Table 3.3) was used in this research to analyze the dispersion and size characteristics of the θ phase (Al_2Cu) precipitate in the matrix. The samples were water quenched to room temperature when the desired aging time was reached. Note that all times listed in Table 3.3 are actual times at temperature. The time to reach temperature was determined experimentally by imbedding a thermocouple in a test coupon. The time to temperature used for each temperature setting is listed in Table 3.2.

TABLE 3.2 SAMPLE TIME TO TEMPERATURE

(°C)	(min)
200	6.75
250	6.50
300	6.75
350	7.25
400	7.50
425	7.67
450	7.75

4. Processing Sequence

In the first stage of this research test coupons were overaged at seven different temperatures ranging from 200 to 450°C for specified times at intervals from 0.2 hrs. up to 500 hrs. No cold work was introduced prior to aging. The process diagram is outlined in Figure 3.2 and actual time interval for each temperature are listed in Table 3.3.

TABLE 3.3 AGING SCHEDULE (NUMBERS INDICATE SAMPLE NUMBER)

TIME (HRS)	0.2	0.5	1	2	5	10	20	50	100	200	500
TEMP (C)											
200	-	1	2	3	4	5	6	7	8	9	10
250	-	11	12	13	14	15	16	17	18	18	20
300	21	22	23	24	25	26	27	26	29	30	31
350	32	33	34	35	36	37	38	38	40	-	-
400	41	42	43	44	45	46	47	48	49	-	-
425	50	51	52	53	54	55	56	57	58	-	-
450	59	60	61	62	63	64	65	70	71	-	-

In the second stage of experimentation cold work was introduced to the samples prior to the aging process to determine its effect on particle size and distribution. This was accomplished by cold rolling and two specific reductions 10% and 20%. The aging of these two different strain conditions was

conducted at three different temperatures, 400, 425, and 450°C. The process is diagramed in Figure 3.3.

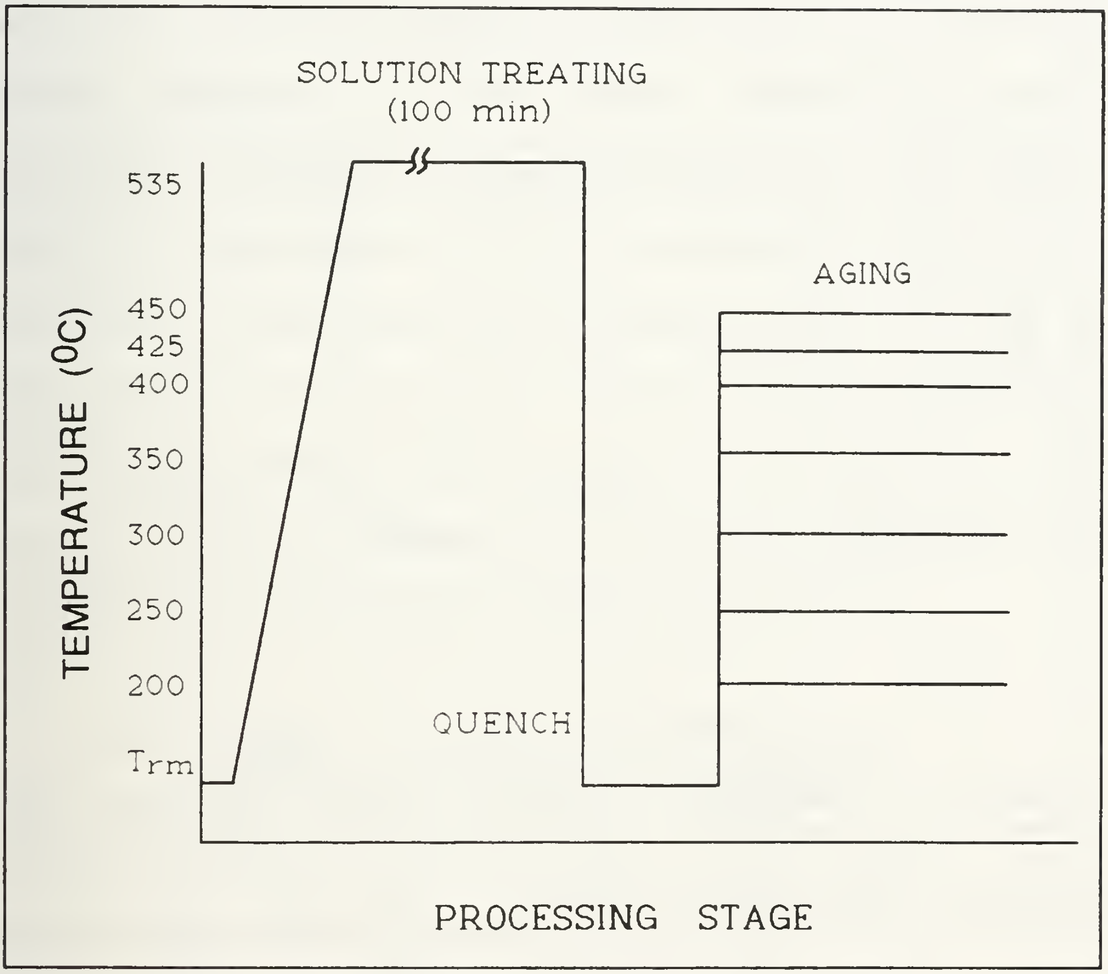


Figure 3.2 Overaging Process Diagram.

C. THERMOMECHANICAL PROCESSING FOR SUPERPLASTICITY

The billets of average size 1.5 x 0.875 x 2.4 in. (38.1 x 22.2 x 61.0 mm) were then processed using a modified TMP schedule developed at NPS for the Al-Mg system. This process is shown in Figure 3.4. It involves solution heat treating the billets at 535°C for 100 minutes [Ref. 25] followed by a room

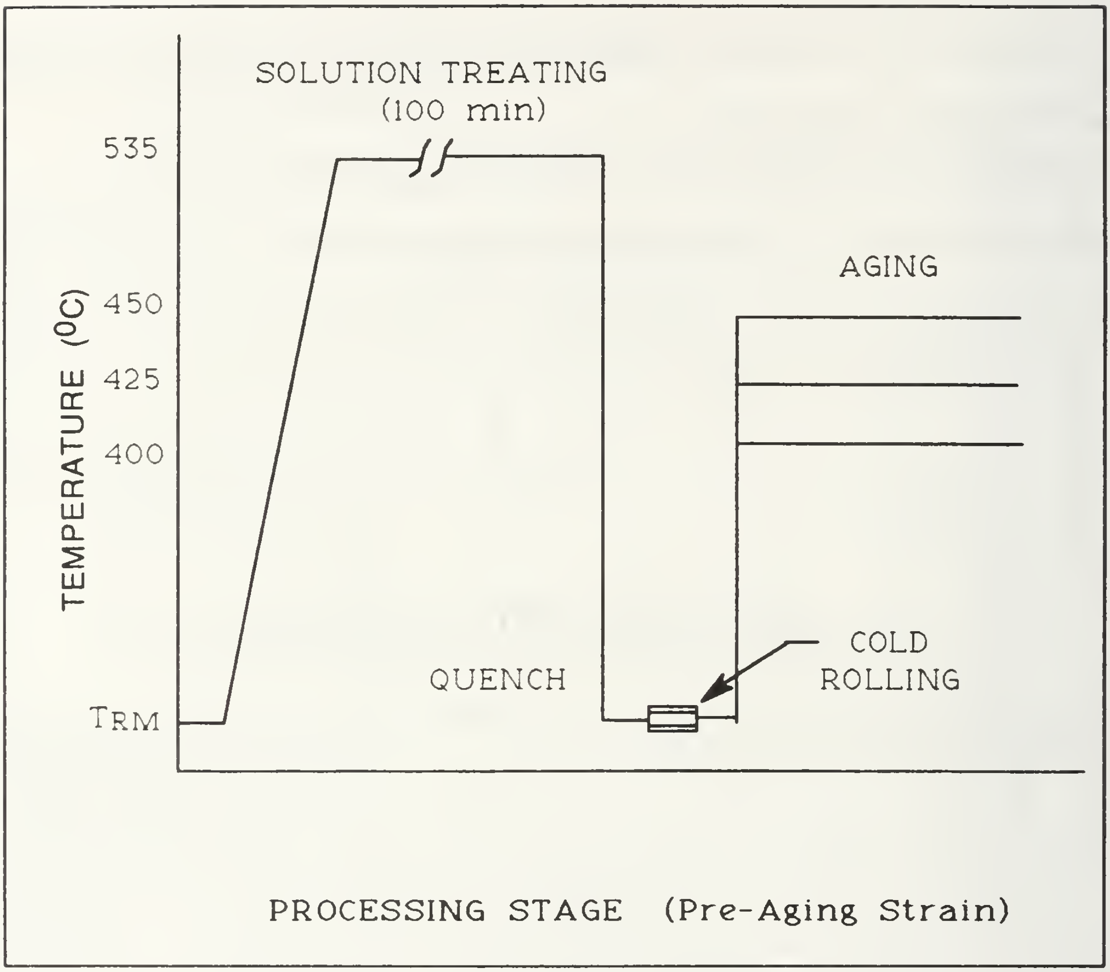


Figure 3.3 Overaging Process Diagram (Initial Cold Work).

temperature water quench. A 10% cold roll was then attempted to form dislocations as nucleation sites for the θ phase precipitates, but severe surface cracking and alligatoring (cracking at the sample ends), due primarily to the small radius rolls of the laboratory mill used, was encountered. The samples were then preheated to 200°C and warm rolled to a 10% reduction in 2 separate 5% rolls. The surface cracking problem was eliminated but some alligatoring was still present in the samples. This was considered a roll

geometry problem and not material related since this alloy is cold rolled in industry to a 7% reduction. The material was then artificially aged for 10 hours at 450°C. This time-temperature combination was determined from the overaging experiments to be the minimum necessary to match the microstructure developed in the superplastic Al-Mg system. After aging the material was furnace cooled at a rate of no more than 5 degrees per minute to one of three test rolling temperatures. The test blocks were then warm rolled at their respective temperatures in nine separate passes of increasing strain rates as shown in Table 3.5, with thirty minutes of reheating between rolling passes. At the completion of rolling, the specimens were quenched in room temperature water. The process is outlined in Figure 3.5.

TABLE 3.5 TMP ROLLING SCHEDULE

PASS	INITIAL THICKNESS (in)	MILL GAP (SET) (in)	FINAL THICKNESS (in)	MILL DEFLECTION (in)	STRAIN (in/in) %	STRAIN RATE (1/s)
1	0.815	0.742	0.766	0.024	6.0	0.8
2	0.766	0.657	0.675	0.018	11.9	1.78
3	0.675	0.581	0.602	0.021	10.8	1.19
4	0.602	0.513	0.530	0.017	11.9	1.33
5	0.530	0.431	0.454	0.023	14.3	1.56
6	0.454	0.338	0.356	0.018	21.6	2.11
7	0.356	0.227	0.25	0.023	29.9	2.86
8	0.25	0.123	0.163	0.04	34.8	3.73
9	0.163	0.045	0.078	0.033	52.1	5.87

The strain induced in each pass was calculated using Equation 3.1.

$$\epsilon=\frac{t_i-t_f}{t_i}=\frac{\Delta t}{t_i} \qquad \text{Equation 3.1}$$

where t_i is the initial pass thickness and t_f is the pass final thickness.

The strain rates were determined from Equation 3.2.

$$\dot{\epsilon}=\frac{2\pi Rn}{\sqrt{Rt_i}}\sqrt{\epsilon}\,(1+\epsilon/4) \qquad \text{Equation 3.2}$$

where R is the radius of the rolls, n is the speed of the rolls in rad/s.

After completion of the processing, samples were sectioned from the material for microstructural analysis and mechanical testing. The pieces designated for microstructural analysis were then annealed for 30 minutes at their respective rolling temperatures.

D. METALLOGRAPHY

1. Scanning Electron Microscopy

The Scanning Electron Microscope was used to study the precipitate (Al_2Cu) size and distribution at various stages of overaging to determine the temperature and time combination, with or without pre-aging strain, required to obtain a microstructure most suitable for superplastic response.

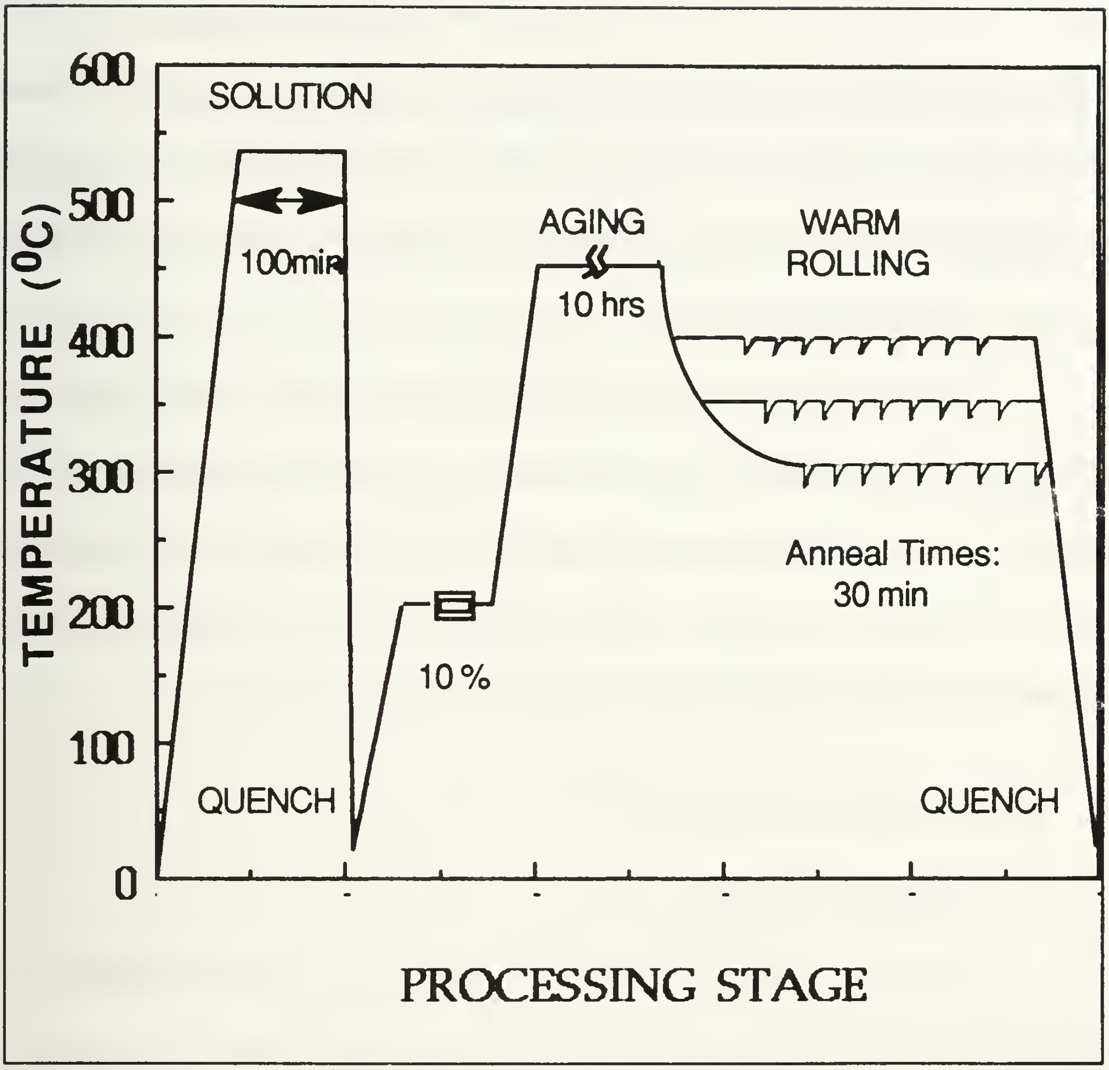


Figure 3.5 TMP Schematic Diagram.

Specimens were first mechanically ground on successively finer grit wet silicon carbide paper finishing with 600 grit. Next, the samples were polished to a scratch-free surface with one micron diamond paste on a polishing wheel. The samples were then electropolished in a solution of 90% Butoxyethanal and 10% HCl maintained at 0°C in a Methanol bath. A constant 16 vdc potential was applied which produced a current of approximately 0.03

amps. The sample was inserted into the electrolytic solution and held parallel to the outer edge of the vortex created by a magnetic stirer. After 3 minutes the sample was removed and washed with methanol. The SEM was used in the backscatter mode with a LaB_6 filament and accelerating voltage of 20 KV.

2. Optical Microscopy

The sample preparation for optical microscopy followed the same pattern as described for SEM, however, an additional anodizing step was included. The anodizing treatment brought out grain boundary contrasts that were not visible in the SEM. This procedure was used on the post TMP processed samples to characterize grain size and structure.

E. MECHANICAL TESTING

1. Hardness Testing

Rockwell hardness tests were conducted on the artificially aged samples immediately after removal from the furnaces. This was done because of the natural aging that occurs in this alloy at room temperature. The hardness data was used to track the extent of softening occurring due to overaging and act as a rough indicator of θ particle size. At least eight indentor tests were performed on each sample and all requirements of reference 28 for valid Rockwell testing was adhered to.

2. Tensile Testing

Tensile testing, to determine the extent of superplastic response achieved, was performed on an Instron Model 6027 test machine with a Marshal Model 2232 clamshell furnace to maintain constant temperature. Tensile test specimens were cut from the sheet produced after a water quench following the final roll of the TMP for each of the three rolling temperatures. See Figure 3.6 for test sample geometry. Several different temperatures and strain rates were used to determine which combination would produce the greatest elongation in the processed material.

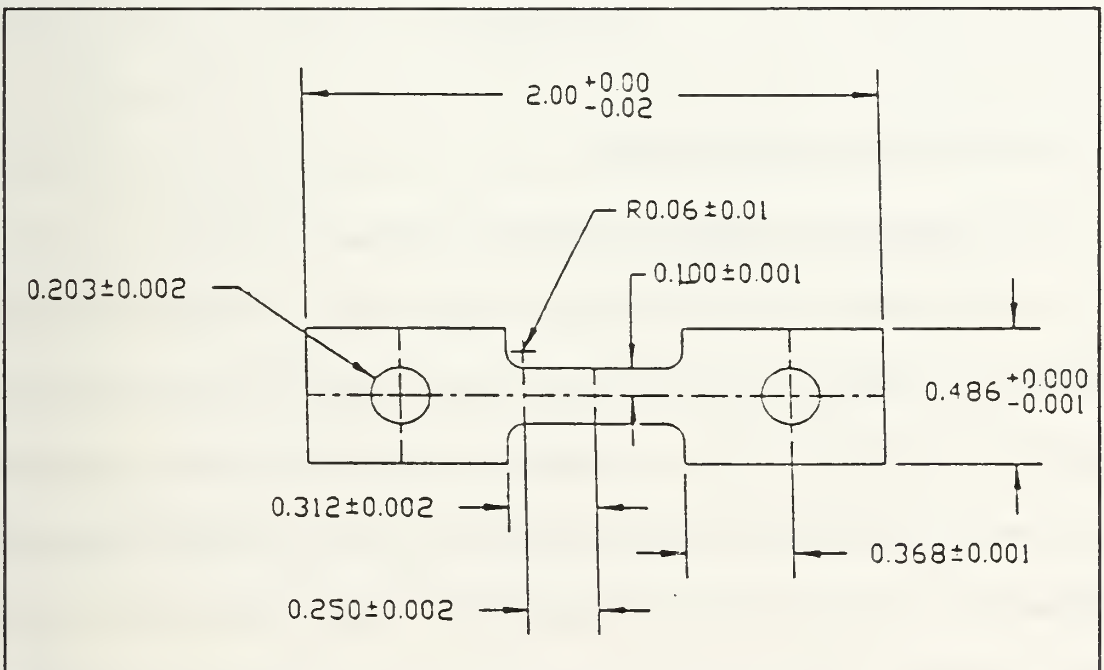


Figure 3.6 Tensile Test Specimen Drawing.

IV. RESULTS

In this chapter, the effects of the overaging process and thermomechanical processing variables on the microstructural development and mechanical behavior of the Al-Cu 2519 alloy will be described. Both SEM and optical microscopy were used to characterize the microstructure of the material during overaging and at various stages of processing. Rockwell hardness tests were used to track the overaging progress and tensile tests conducted to assess processing effects on the materials' ductility.

A. EFFECT OF OVERAGING

1. Mechanical Property (Hardness Data)

Hardness data reflecting the overaging response of the Al-Cu 2519 commercial grade alloy is presented graphically in Figures 4.1 and 4.2. It should be noted that the times and temperatures used to produce these data far exceed what is used to age harden this alloy for normal engineering applications. This experiment was specifically designed to take the material into a severely overaged condition in an attempt to exploit the θ phase precipitate particles as sites for PSN during subsequent processing steps. In Figure 4.1 Rockwell Hardness 'B' scale was used to track the response of samples aged at five different temperatures varying from 200°C to 400°C for

times up to 500 hours. Of particular note is the large drop in hardness observed when aging at 400°C compared to 350°C and below. In Figure 4.2 the hardness scale was changed to the Rockwell 'F' scale to obtain readings on the samples in this very soft condition. The low hardness when aging at 400°C and above is indicative of the change from the θ' to θ (Al_2Cu) phase [Ref. 27]. In addition, it is observed that the rate of change in hardness with aging time becomes less after about 10 hours at 425°C and 450°C. The slightly increased hardness upon aging at 450°C is believed due to higher equilibrium of copper content in the solid solution at the higher temperatures and thus a contribution of solid solution strengthening.

After completion of these initial aging experiments pre-aging strain was introduced to a new batch of solution treated specimens in an attempt to accelerate precipitation and enhance the θ-phase distribution. Aging tests were run at only the three highest temperatures 400, 425 and 450°C and for a time of up to 100 hours. Figure 4.3 tracks the hardness decrease in the 10% strained samples on the RHF scale. Figure 4.4 shows the RHF readings for the 20% cold rolled material. Comparison of the data of Figures 4.3 and 4.4 to those of Figure 4.2 reveals nearly identical aging response and thus little acceleration due to the pre-strain upon aging at these relatively high temperatures.

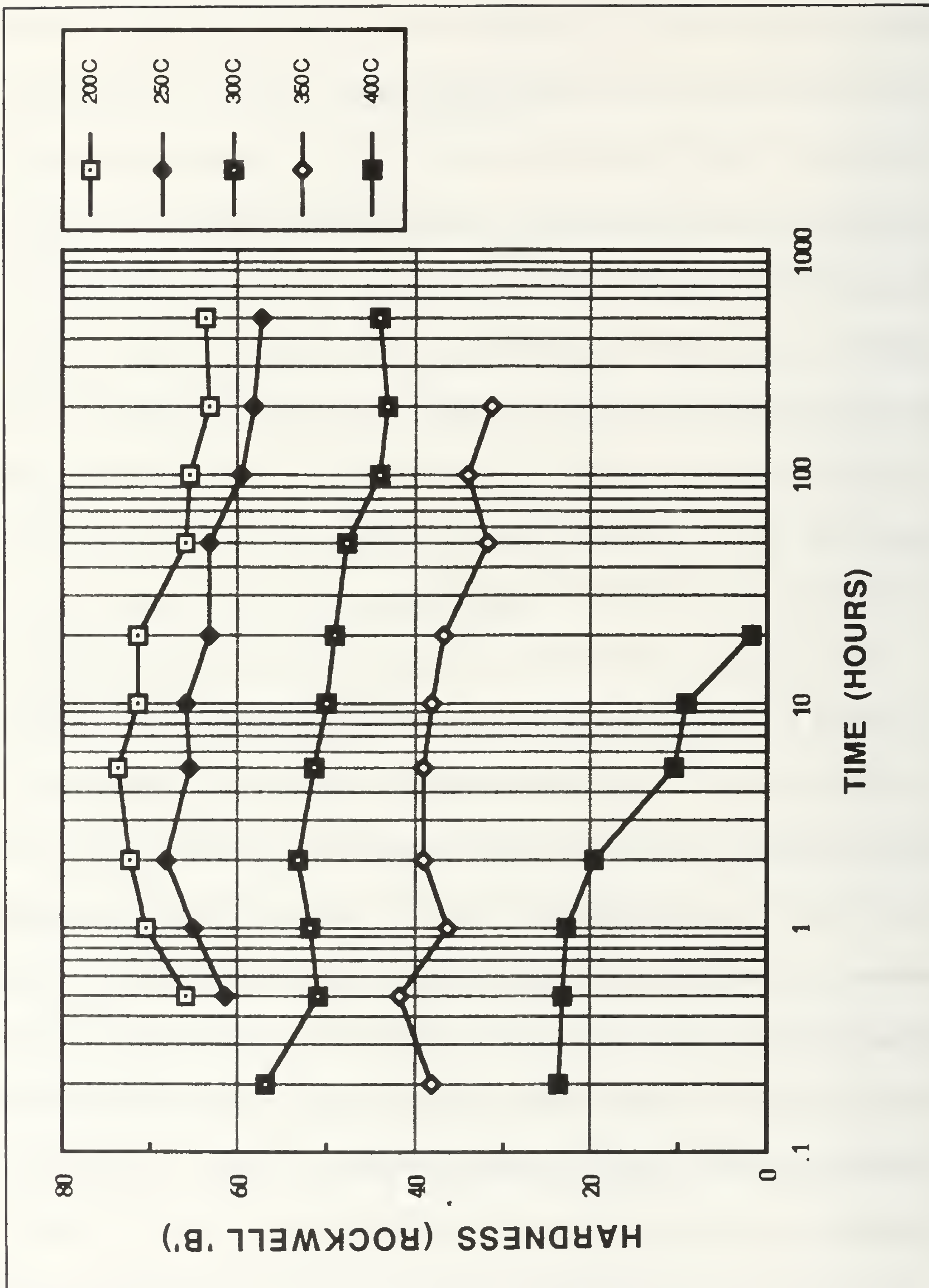


Figure 4.1 Age Hardening Response (Intermediate Temperatures).

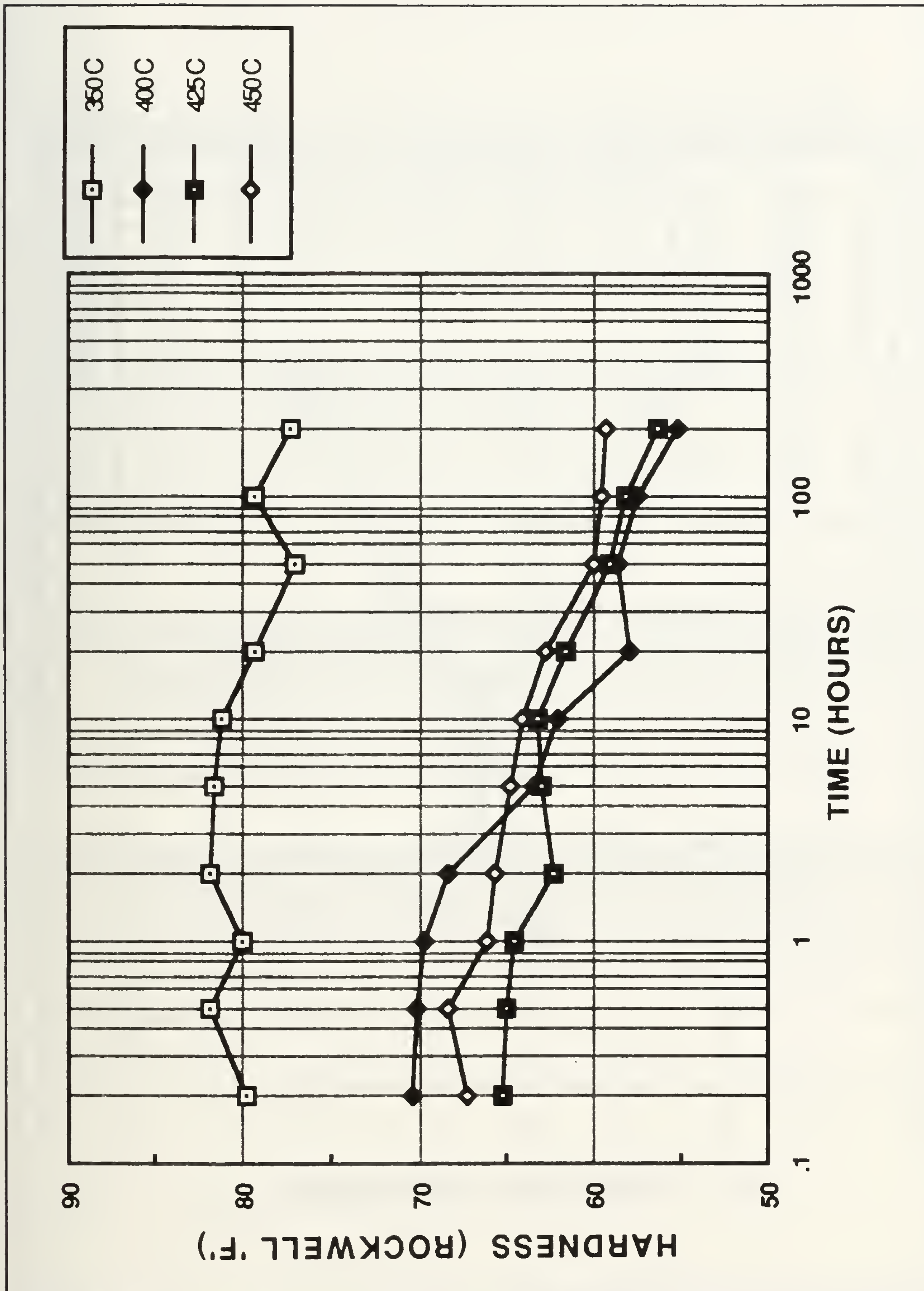


Figure 4.2 Age Hardening Response (High Temperatures).

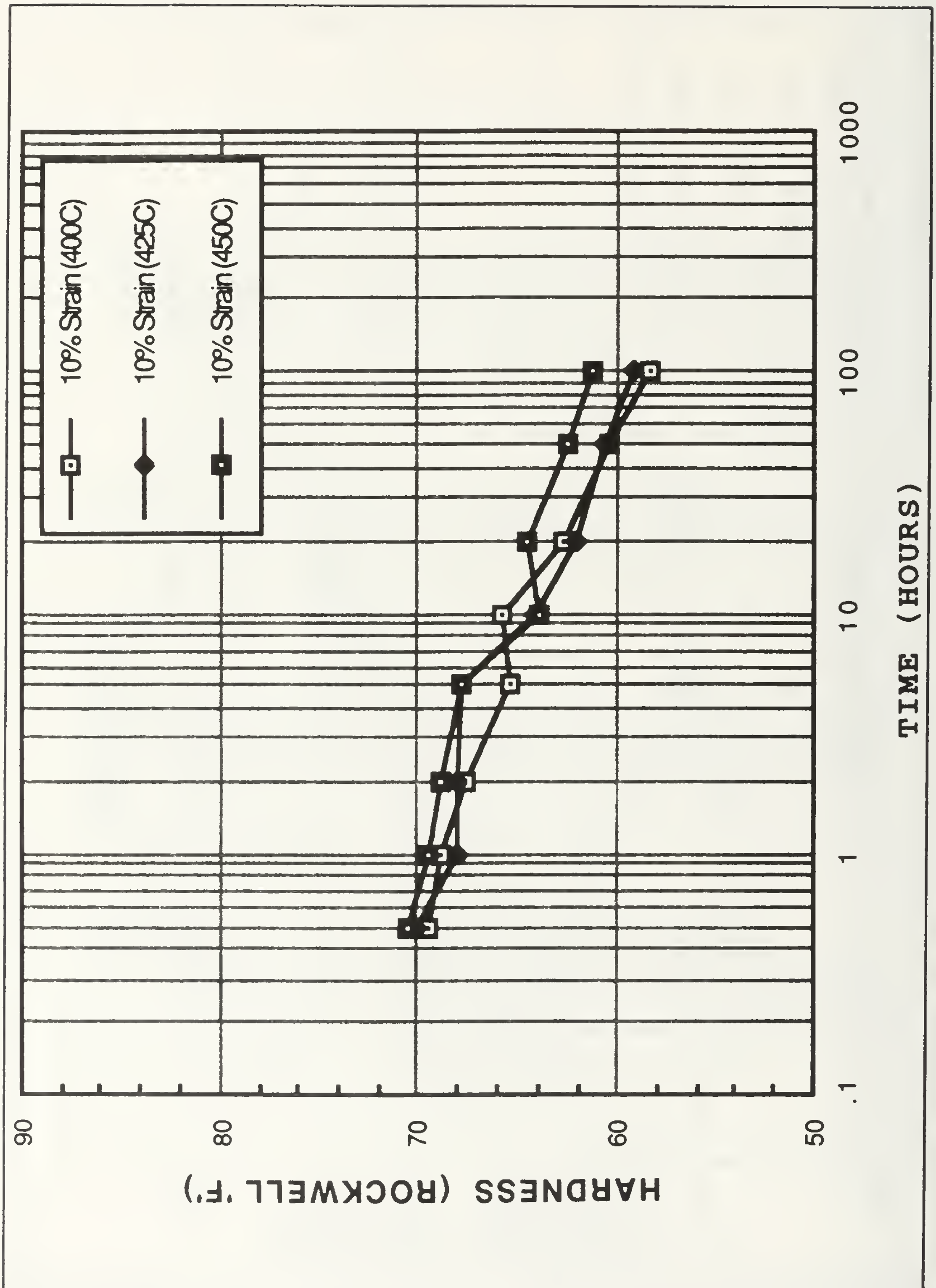


Figure 4.3   Age Hardening Response (10% Strain).

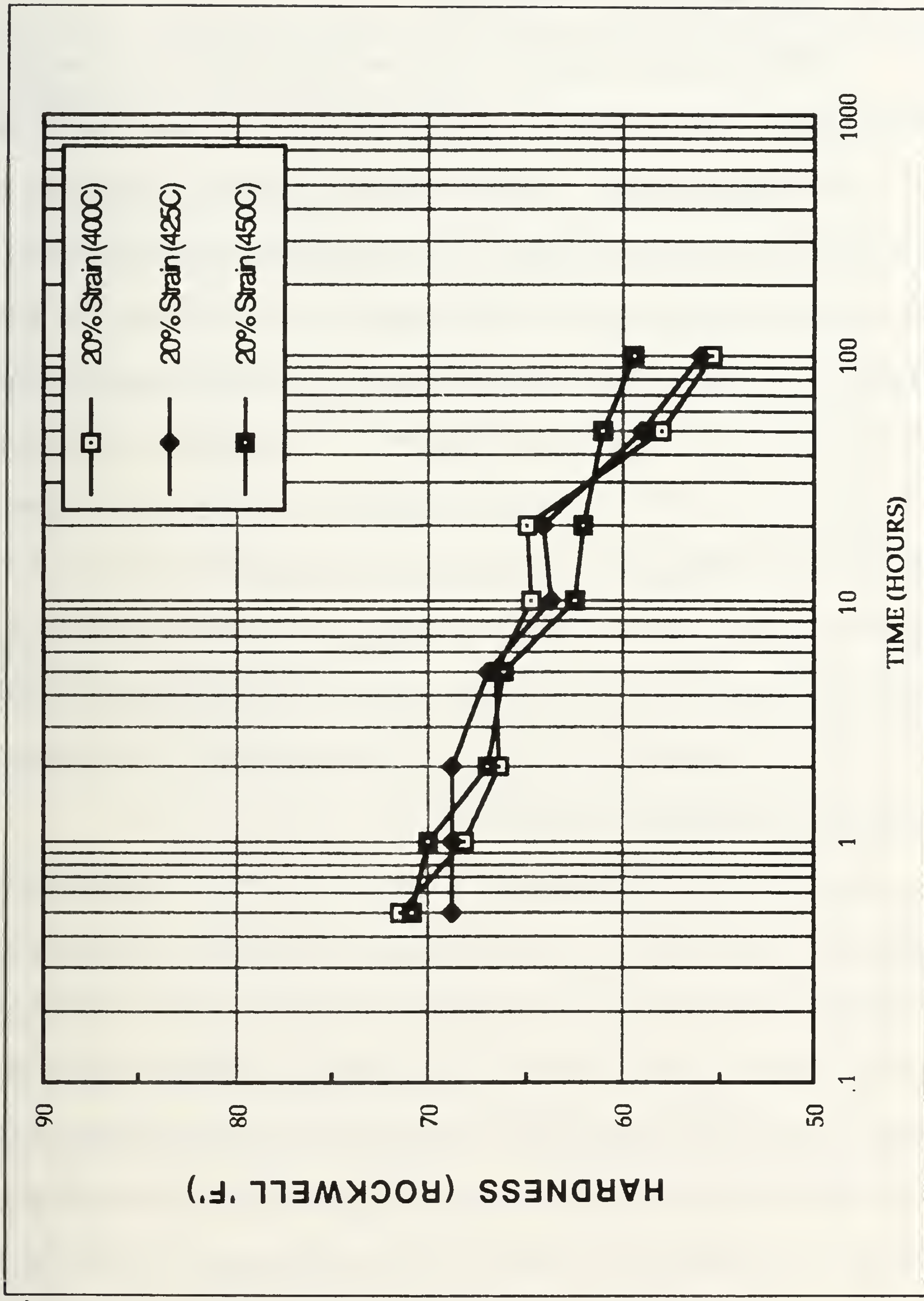


Figure 4.4   Age Hardening Response (20% Strain).

2. Microstructure (SEM Data)

Microstructural analysis of the test samples was then conducted using the SEM, in the backscatter mode, to determine the θ phase particle size and distribution at various overaged conditions. Figure 4.5 (a) shows the microstructure after one hour at 400°C and Figure 4.5 (b), one hour at 450°C. The white particles are θ phase (Al_2Cu) precipitates in a dark α matrix. Little difference is attributable to aging temperature at this short time and in both case the predominant particle size is less than 0.5 micron. Many particles are elongated with an aspect ratio of about 2.0. In the next set of micrographs, Figure 4.6, the sample time at temperature was increased to ten hours and increased particle size is especially apparent in the 450°C sample. The particles now average about 1.0 micron in size. At 50 hours, Figure 4.7, the size continues to increase to about 1.5 microns but there are still numerous smaller particles apparent in the matrix.

The effects of 10% pre-strain on the particle size and distribution are illustrated in Figure 4.8. For the micrographs of Figure 4.9, 20% strain was introduced before aging. In both cases the particle size has increased and particles are more nearly equiaxed. Examination of numerous areas also suggests a more uniform distribution due to pre-strain. The particle size in the 450°C sample is now 2.0 microns. Particles of 1.0μm size have been associated with PSN of recrystallization during TMP of the Al-Mg alloys. Thus the 450°C,

10%, pre-strained sample, aged for ten hours was then chosen as a starting condition for the TMP of this research.

B. EFFECT OF THERMOMECHANICAL PROCESSING

The thermomechanical processing technique developed for the Al-Mg system, which was described in detail in the procedure section of this report, was then run on the material using as a start point for the warm rolling step the condition described in the above paragraph.

1. Microstructure (SEM/Optical)

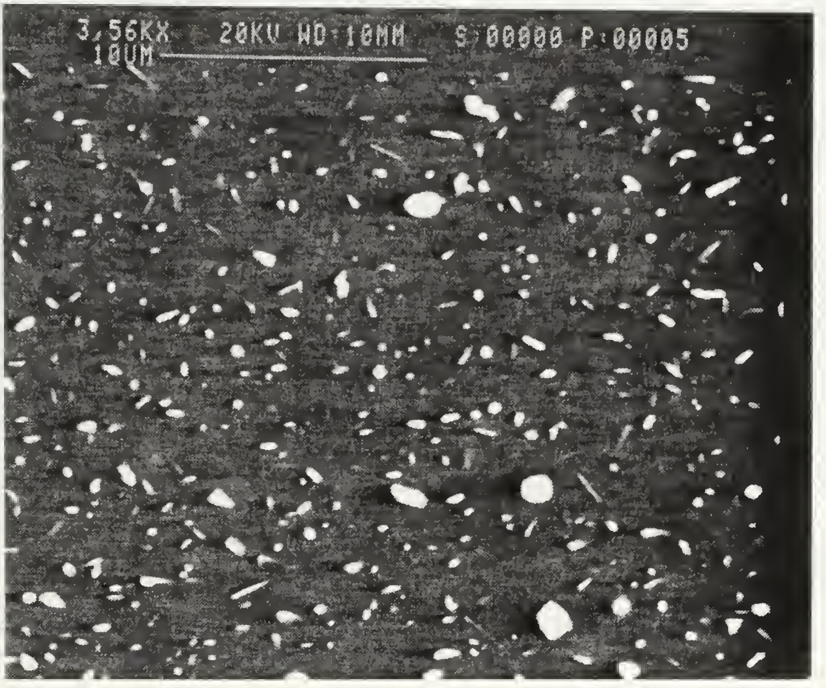
Figure 4.10 shows the θ particles upon completion of the TMP using a 400°C warm rolling temperature and after a 30 minutes, 400°C anneal following the completion of rolling. Both the size and distribution of the particles is consistent with overaging experimental results.

Optical microscopy was then utilized to characterize the grain size and structure. Attempts to bring out the grain structure on the SEM using several different polishing techniques were unsuccessful. Optical micrographs, Figure 4.11, show the grain structure just before start of the TMP (a), immediately following the quench after the 400°C rolling (b), and after a 30 minute post-roll anneal, also at 400°C (c). The grain size appears to be reduced by the TMP. The large, irregular grains prior to processing have been replaced by a fine, more equiaxed structure. The resulting fine grains are on the order of 20 to 30 microns.

2. Superplastic Response (Tensile Test Data)

Initial tensile tests conducted on the TMP processed material show superplastic response was just obtained (~205%). Figure 4.12 shows percent elongation obtained for the three separate TMP roll temperatures when tested at a constant strain rate and various temperatures ranging from 300 to 450 C. A maximum elongation of 205% was achieved on the sample rolled at 350 C in the TMP and then pulled in a tensile test at a strain rate of 6.7×10^{-4} s^{-1} at 450 C.

(a)



(b)

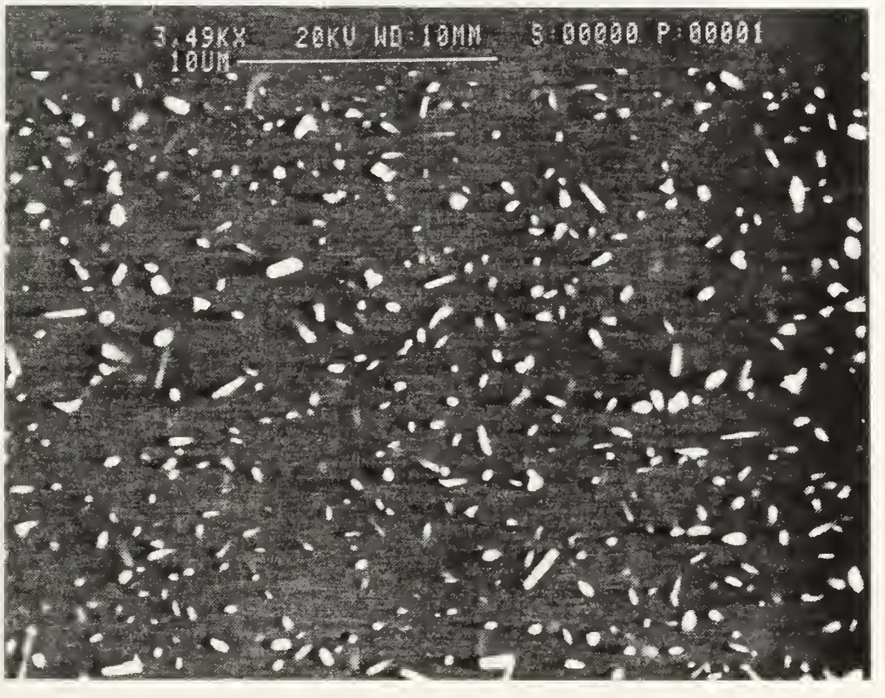
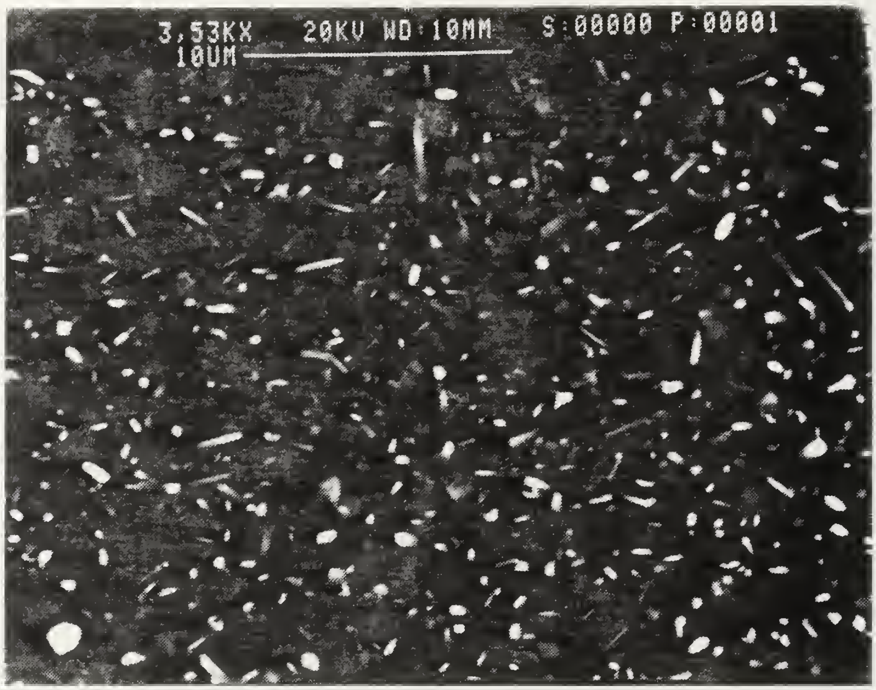


Figure 4.5 **Backscattered SEM micrograph showing the Al-Cu 2519 alloy after aging for 1 hour. (a) 400°C; (b) 450°C.**

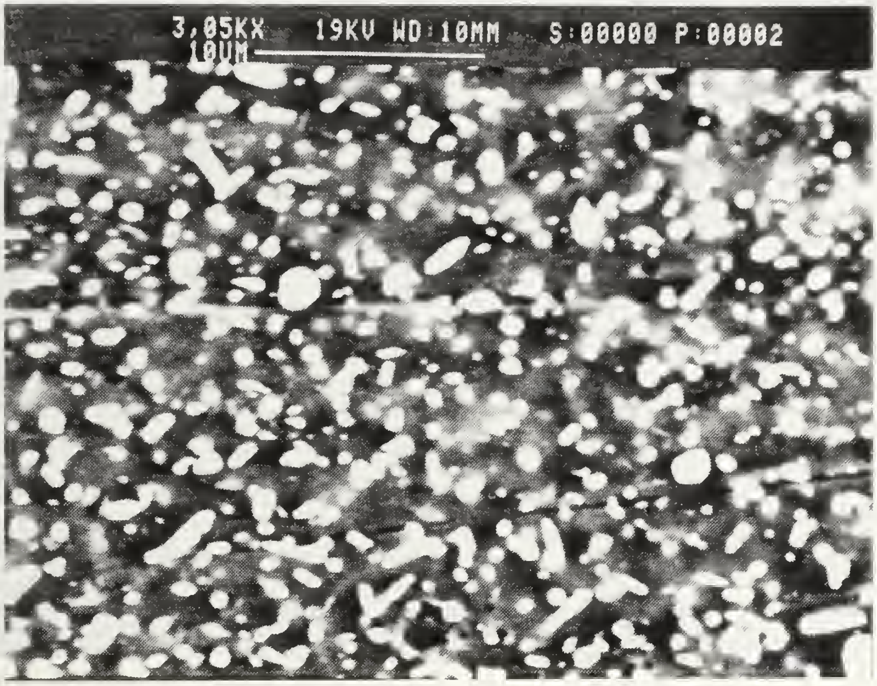
(a)



(b)

Figure 4.6 Backscattered SEM micrograph showing the Al-Cu 2519 alloy after aging for 10 hours. (a) 400°C; (b) 450°C.

(a)



(b)

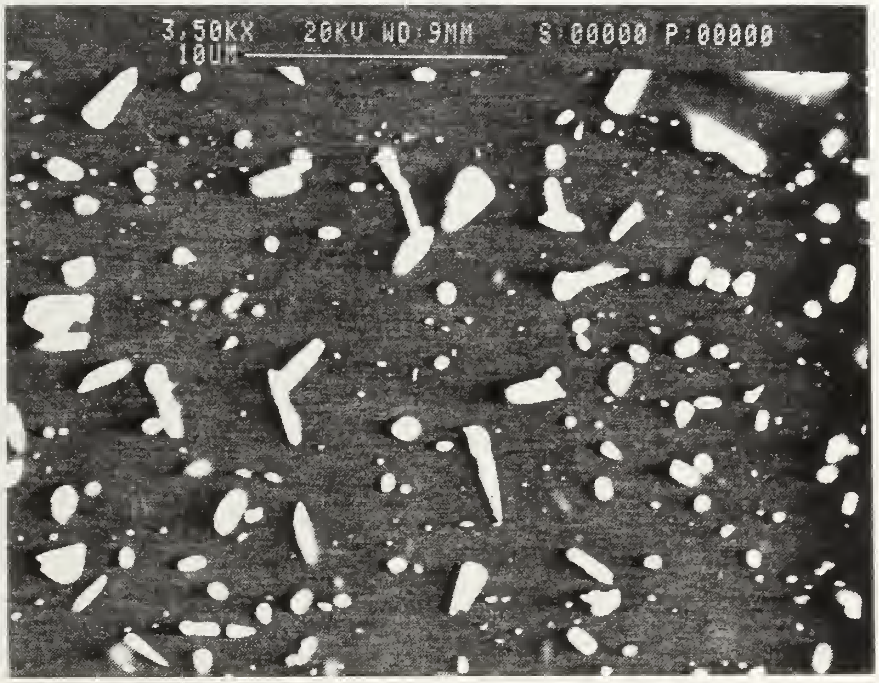


Figure 4.7 Backscattered SEM micrograph showing the Al-Cu 2519 alloy after aging for 50 hours. (a) 400°C; (b) 450°C.

(a)

(b)

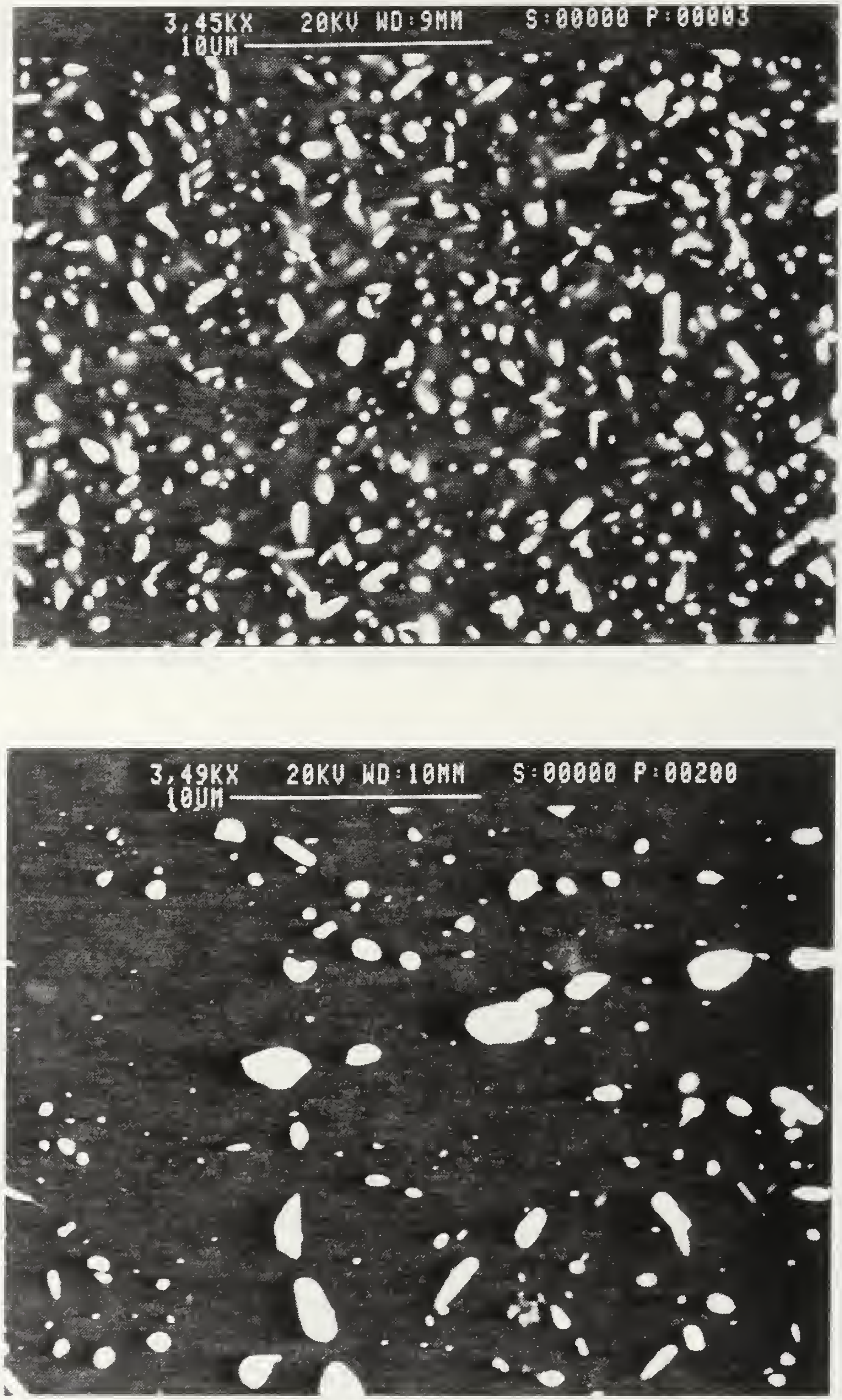
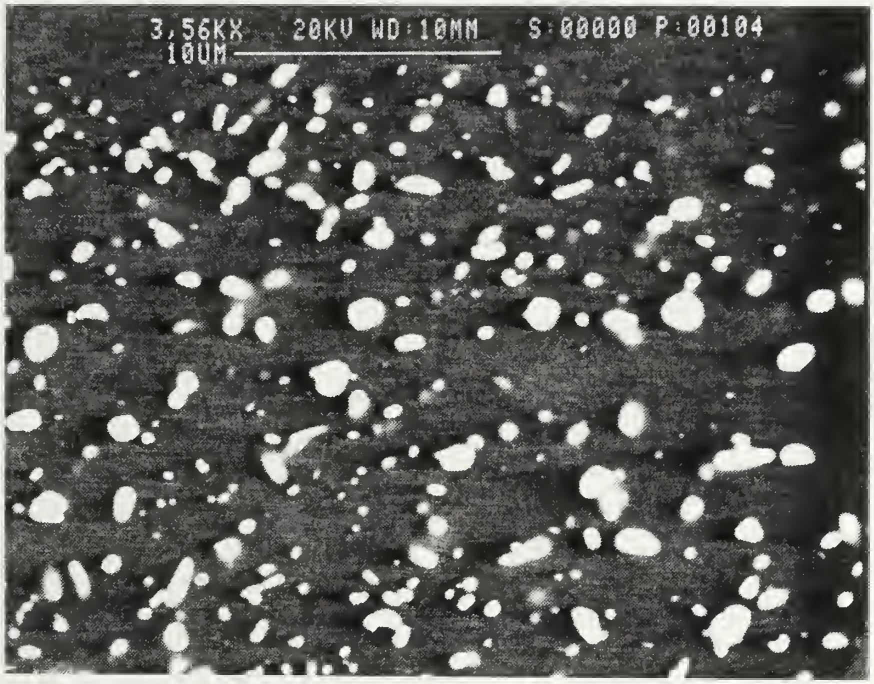


Figure 4.8 Backscattered SEM micrograph showing the Al-Cu 2519 alloy after aging for 10 hours, 10% strain. (a) 400°C; (b) 450°C.

(a)



(b)

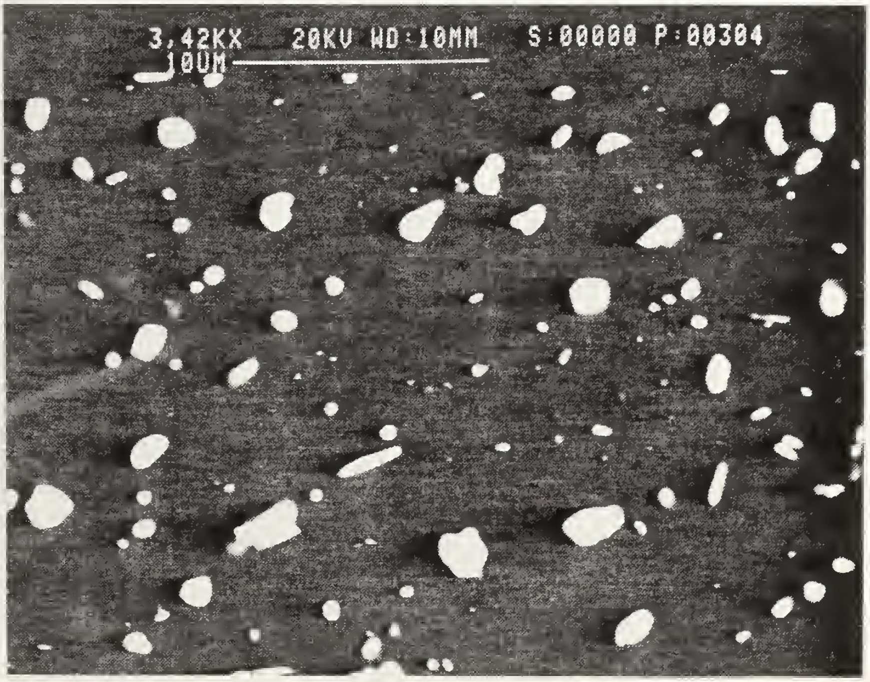
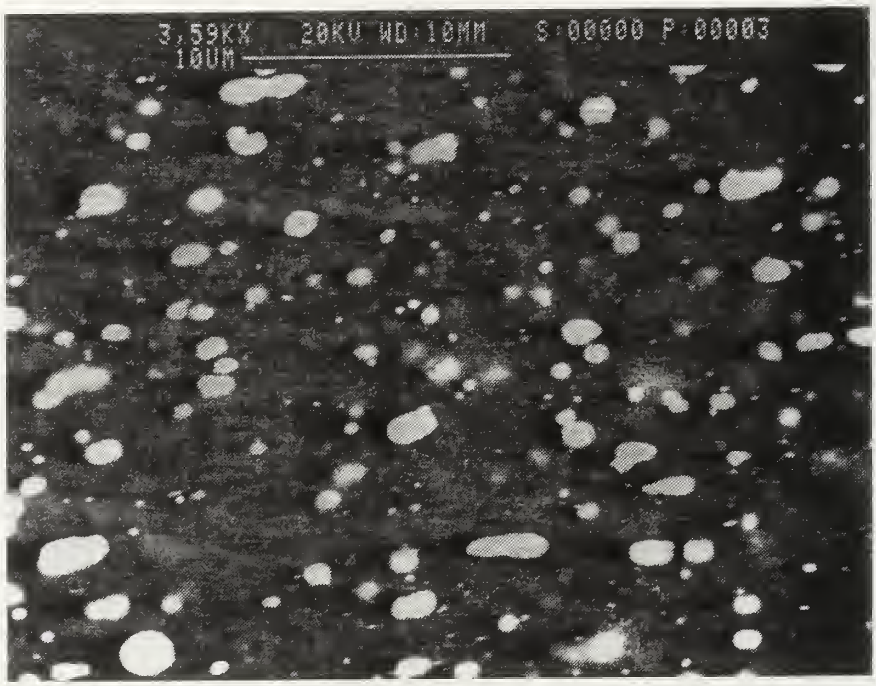


Figure 4.9 **Backscattered SEM micrograph showing the Al-Cu 2519 alloy after aging for 10 hours, 20% strain. (a) 400°C; (b) 450°C.**

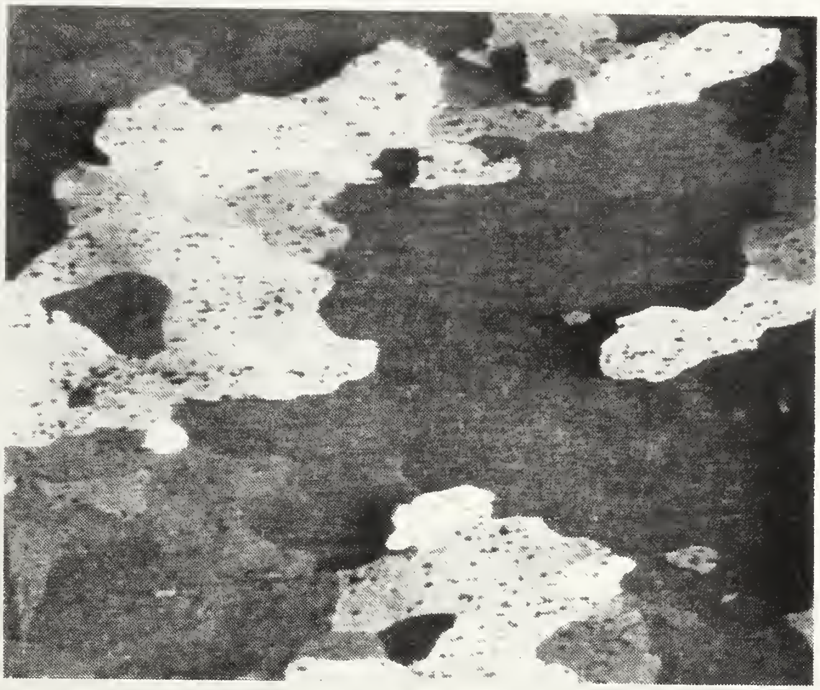
(a)



(b)

Figure 4.10 **Backscattered SEM micrograph showing the Al-Cu 2519 following TMP process 400°C roll. (a) no post-roll anneal; (b) 30 min. post-roll anneal.**

(a)



(b)

Figure 4.11 Optical micrographs of Al-Cu 2519 alloy, 400°C TMP (100X). (a) post-age, pre-roll condition; (b) post-roll, quenched condition (continued..);

(c)

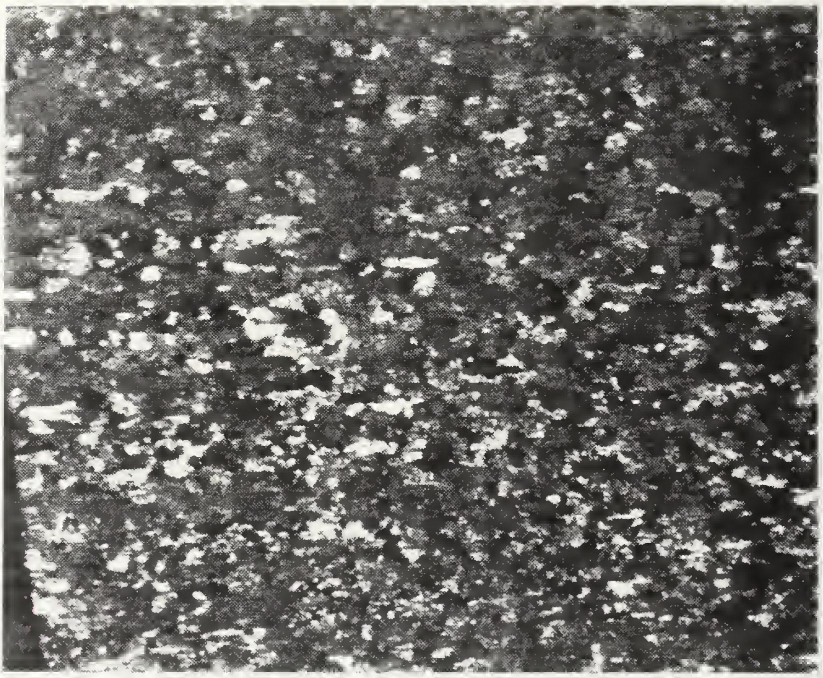


Figure 4.11 (c) 30 min., 400°C post TMP anneal.

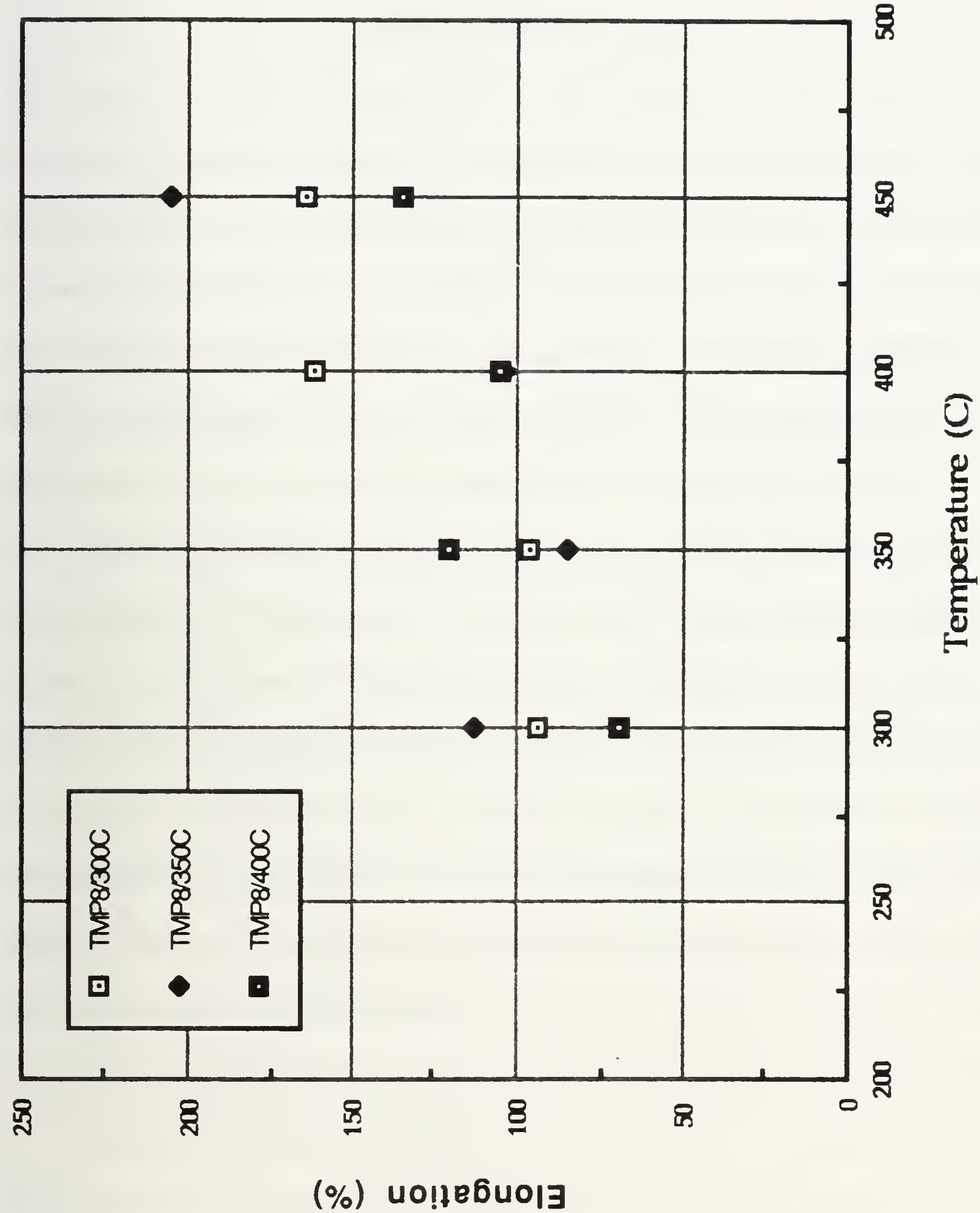


Figure 4.12 **Tensile Test Results** ($\dot{\varepsilon}=6.7\times10^{-4}S^{-1}$)

V. DISCUSSION

This study of extreme overaging on Al-Cu 2519 material has developed data of the temperature-time-growth relationship of the θ phase (Al_2Cu) precipitate. This information is necessary as a starting foundation for developing a processed microstructure in the 2519 alloy similar to the superplastic microstructure in the Al-Mg system. The essential feature of the NPS processed superplastic Al-Mg structure is PSN occurring at the β phase particles during annealing in the latter stages of the TMP [Ref. 7]. The overaging studies showed clearly that a θ phase particle of sufficient size to promote PSN would not be produced during a 300°C anneal process alone, as achieved in the Al-Mg system. In an overaging study conducted on 2219 by Willig and Heimendahl it was reported that, at 350°C, the θ' phase is no longer stable and transforms to the equilibrium θ phase [Ref. 27]. Our results show a substantial decrease in hardness on samples aged at temperatures above 350°C, which supports this transition to θ upon aging above this temperature. Therefore it was determined that a temperature at least above 400°C is required to produce θ particles of the desired size in a practical time.

In addition to size the particle distribution was also considered. In the 2xxx series Al alloys, cold working of freshly quenched material greatly

enhances its precipitation heat treatment response [Ref. 25]. Therefore, test samples were given an initial cold roll to 10% and 20% before artificial aging. The results from SEM analysis show a better distribution of the θ phase and an increase in particle size in the pre-worked material, but the very slight differences between the 10% and 20% indicates a 10% cold roll is sufficient to enhance distribution.

Optical microscopy results show grain refinement through the TMP was achieved after a 30 minute post anneal heat treatment. Attempts at better imaging of the structure utilizing the SEM were unsuccessful.

Initial mechanical testing results show limited superplastic behavior was obtained, and the ductility in the material was increased from a published value of 50 to 75% at elevated temperatures, depending on temper, [Ref. 22] to 205% elongation. It should be noted that this thesis represents only the preliminary work in developing a thermomechanical process which will render the commercial grade 2519 alloy superplastic. Additional microstructural analysis of the processing phases is required and adjustments to the TMP made as indicated by these results.

V. CONCLUSIONS

The following conclusions are drawn from this research:

1. A relatively high temperature, above 350°C, is required to produce the stable θ phase (Al_2Cu) precipitate in this Al-Cu 2519 alloy.

2. The size of the second phase θ precipitate is dependant on the aging time and temperature. Its distribution can be improved by pre-straining the alloy prior to aging.

3. The hardness of this alloy, in the overaged condition where θ phase is produced (temperatures above of 350°C [Ref. 27]), decreases with increased aging time and temperature. If the temperature is increased above approximately 400°C and the material aged for over 10 hours the hardness does not drop off as sharply. This is a result of a higher copper content in the solution and thus solid solution strengthening is occurring.

4. An overaging time of ten hours at 450°C with 10% strain produces a precipitate of the approximate size (1.5μm) and distribution in this alloy deemed desirable for PSN during superplastic processing.

5. The modified Al-Mg TMP schedule enhances the ductility of the 2519 Al-Cu alloy.

6. A refined microstructure was obtained following TMP.

VI. RECOMMENDATIONS

The following are recommendations for further study:

1. Conduct more extensive tensile testing on the three temperatures of TMP processed material produced in this experiment. Use a variety of strain rates and temperatures to accomplish this.

2. Develop and utilize scanning electron microscopy techniques to better assess the effect of the TMP warm rolling parameter differences on the microstructure.

3. The following are suggested changes to the TMP process to achieve the most optimum microstructure for superplastic response in the 2519 alloy:

   - Increase the 450°C pre-rolling aging time, to increase particle size.
   - Reduce the warm rolling temperature.
   - Vary the anneal time between rolls.

4. Use the material as received, in the T87 temper, as a start for the process sequence. Thus, the solution treatment and cold roll steps of the process could be eliminated.

5. Investigate thoroughly the mechanical properties of both the β and θ phase particles to better determine their effectiveness in PSN.

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